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**A STUDY OF TERNARY PHASE DIAGRAMS  
OF TUNGSTEN AND TANTALUM**

*W. Rostoker*

*Armour Research Foundation of  
Illinois Institute of Technology*

MARCH 1960

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# **A STUDY OF TERNARY PHASE DIAGRAMS OF TUNGSTEN AND TANTALUM**

*W. Rostoker*

*Armour Research Foundation of  
Illinois Institute of Technology*

*MARCH 1960*

Materials Laboratory  
Contract No. AF 33(616)-5678  
Project No. 7351

WRIGHT AIR DEVELOPMENT DIVISION  
AIR RESEARCH AND DEVELOPMENT COMMAND  
UNITED STATES AIR FORCE  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

## FOREWORD

This report was prepared by Armour Research Foundation under USAF Contract No. AF 33(616)-5678, Project Nr. 7351 "Metallic Materials", Task Nr. 73512 "Refractory Metals". It was administered under the direction of the Materials Laboratory, Wright Air Development Center, with Captain R. M. Quimby acting as project engineer.

This report covers the period of work from 1 April 1958 to 31 July 1959.

Armour Research Foundation personnel who made major contributions to this program were: W. Rostoker and A. G. Metcalfe, project directors; R. F. Domagala, project engineer; A. W. Goldenstein, project engineer; A. Siede, project engineer; J. R. Dvorak, metallographer; R. A. Moll, technician; J. R. Wright, technician. The data are recorded in Logbooks Nos. C-7878, -8109, -8200, -8497, -8680, and -9100, assigned to ARF Project 2145. This final report has been designated internally as 2145-15.

ABSTRACT

Phase relationships in 36 ternary refractory metal systems were investigated. Some 28 related binary systems were first established by reviewing the literature and doing whatever supplementary experimental work necessary to assure a valid basis for the ternary studies.

Nearly 200 ternary alloy compositions were prepared from high-purity alloying ingredients by nonconsumable electrode arc-melting procedures. These alloys were studied metallographically in the as-cast condition as well as after annealing at and quenching from 1500 and 1000°C.

The main purpose of this effort was to scan the solid solubility limits of ternary systems based on tungsten and/or tantalum and involving the following metals: molybdenum, niobium, vanadium, chromium, columbium, and rhenium. The diagrams presented herein are intended to serve as a foundation for future alloy development. Because none of the systems presented is completely detailed, definitive work can and should be done as the specific need arises.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

*I. Perlmutter*

I. PERLMUTTER  
Chief, Physical Metallurgy Branch  
Metals and Ceramics Division  
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# A STUDY OF TERNARY PHASE DIAGRAMS OF TUNGSTEN AND TANTALUM

## I. INTRODUCTION

It has been the purpose of this research program to define the phase relationships in a large number of ternary systems based on tungsten and tantalum in detail sufficient for the purposes of exploratory alloy development. It was felt that alloy development needed information primarily on the extent of terminal solubility of solid solutions based on tungsten and tantalum. Secondly, there would be interest in the potentialities of age hardening as indicated (a) by changes in solid solubility with decreasing temperature, (b) in the nature of solidification reactions if these were unusual, and (c) in the identity of intermetallic compounds entering into equilibrium with the terminal solid solutions.

The philosophy in approaching this work has been that relatively few alloys intelligently selected, whose structures are properly interpreted, can reveal the aforementioned points of information with sufficient accuracy to serve the interests of alloy development. The value of this modus operandi lies in the large number of systems which can be studied with a modest expenditure of effort and funds. It is expected that as the metallurgy of tungsten and tantalum matures, the need will arise for detailed and highly accurate definitions of phase equilibria. This work should be done at that time when the need has been defined.

The systems studies contain elements which melt at 1900°C or higher. In all, six metals--Mo, Nb, V, Cr, Os, and Re--were nominated to be associated with tungsten and/or tantalum. Elementary calculations reveal that some 36 ternary systems must be included in the program. The constitution of the ternary systems was to be defined in terms of isothermal sections at 1500°C and 1000°C. This represents a convenient span of temperatures for indication of potential age-hardening capabilities.

It will be appreciated that studies of ternary diagrams must be based on adequate knowledge of the associated binary systems. There are 28 binary metal systems possible by all combinations of the eight metals used in this program. Nearly all of these are pertinent to the objectives outlined. Of these, eleven systems were not known or were inadequately described by existing published work. It was necessary to add to the program some studies of these systems.

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Altogether about 180 alloys were produced by nonconsumable electrode, cold mold arc melting. Alloys were annealed at the aforementioned temperatures of 1500°C and 1000°C for sufficient time to give equilibrium or a close approach to it. Each specimen in the as-cast or annealed state was examined metallographically. In cases where additional information was necessary, X-ray diffraction patterns were taken and interpreted.

From many years of experience with this type of work it has been found that correction of nominal compositions by weight loss after melting is a simple and reasonably accurate method of defining alloy composition--provided, of course, that the initial ingot charge is accurately weighed on an analytical balance. Only alloys containing vanadium or chromium showed significant weight losses. In general, weight losses on ten-gram button melts not containing either of these two elements were less than 0.020 grams. Since almost all alloys contained at least 5 weight per cent of each component, this represents a limiting error of about 0.5 per cent. This accuracy is considered more than sufficient for present purposes. Those alloys containing vanadium and especially chromium were corrected on the basis that all weight losses were of these elements. Compositions indicated in this report are thus probably accurate to better than one weight per cent. In the two cases where vanadium and chromium were jointly present, composition corrections were based on the experience with relative volatility gained from the other alloys.

## II. EXPERIMENTAL PRACTICES

All of the alloys were produced by melting in a nonconsumable electrode, cold mold, arc-melting furnace operating under a small positive pressure of argon. In general, ten-gram button-shaped ingots were produced. It was invariably necessary to remelt up to ten times to ensure complete fusion and homogeneous alloying of the components. As mentioned earlier, careful measurement of the weight losses incurred by volatilization during melting was used to correct the nominal composition.

Serious difficulty was encountered in producing alloys containing vanadium and especially chromium. The difficulties were of two types: inability to effect complete melting and excess volatilization. When pure elements were used as melting charges, it was found that the heavier metals, particularly tungsten, tantalum, osmium, and rhenium, tended to settle to the bottom of the molten pool and remain in the form of incompletely melted or dissolved pieces. Repeated crushing and remelting did not circumvent this problem and aggravated the weight losses.

One of the methods used to assist in obtaining homogeneous melts was to lay the charge in the crucible so that the tungsten or tantalum, for instance, was disposed on top of the lower melting point metals. In this way the arc preferentially melted the higher melting point metal, which then ran down

over the chromium (for instance) and melted it immediately. It was found that intersolution of the two melts occurred more efficiently. This practice was particularly useful in preparing master alloys containing about 80-90% tungsten or tantalum.

The appropriate use of master alloys instead of pure components was found to be mandatory. This derives from two considerations: By diluting the volatile component, its vapor pressure is reduced and subsequent weight losses are minimized. Secondly, by choosing a master alloy composition with a considerably raised melting point, the problem of incomplete melting was minimized. It will be seen from known phase diagrams that often 60% of tungsten or tantalum is needed in a master alloy to increase the melting point appreciably. Master alloys of chromium with tungsten in the range of 60-80% W are particularly difficult to make but necessary for certain of the ternary compositions.

The highest purity alloying elements available commercially were used in the preparation of these alloys. The stated purities and origin are summarized in Table I.

Attempts were made to improve purity by having the elements melted in an electron-bombardment melting unit operated by a commercial organization. The vanadium purity was not improved. The weight loss of the osmium proved prohibitive in view of the price. Delays in handling certain other elements caused so much lost time that electron bombardment melting was not employed for purifying osmium, rhenium, or chromium.

All alloy ingots were crushed into large fragments and the fractured surfaces examined visually for signs of incomplete melting. One fragment was mounted for a metallographic appraisal of the cast structure and a recheck of incomplete melting.

After a standard homogenization pretreatment of one hour at 1700°C, fragments of each melt were annealed at 1500°C for about 45 minutes to approach equilibrium. In some cases this time was not sufficient to eliminate coring completely, but a standard heat treatment had to be established to handle the host of specimens. Specimens were annealed in evacuated Vycor bulbs at 1000°C for 100 hours after the prior homogenization heat treatment at 1700°C. The 1700° and 1500°C treatments were performed in an evacuated, multi-radiation shield, resistance furnace with a molybdenum tubular resistance element. The specimens were suspended in a basket inside the tubular element. Quenching was performed by dropping the specimens on a water-cooled copper hearth. Temperatures were controlled and monitored with the aid of a manually operated optical pyrometer. Control was generally of the order of  $\pm 25^\circ\text{C}$ . The 1000°C anneals were performed in conventional resistance furnaces controlled automatically to  $\pm 3^\circ\text{C}$ . Specimens were quenched by crushing the Vycor bulb as withdrawn from the furnace in a pail of water.

Specimens were examined primarily by metallography but also by X-ray diffraction where deemed of added value. Metallographic polishing techniques

TABLE I  
FORM, PURITY, AND SUPPLIER OF ALLOYING ELEMENTS

| Element     | Form         | Nominal<br>Purity<br>(%) | Supplier                               |
|-------------|--------------|--------------------------|--|
| Tungsten*   | Powder       | 99.9+                    | Fansteel Metallurgical Corp.           |
| Tungsten    | Rod          | 99.9+                    | Jarrell-Ash                            |
| Tantalum*   | Powder       | 99.9+                    | Fansteel Metallurgical Corp.           |
| Molybdenum* | Powder       | 99.9+                    | Fansteel Metallurgical Corp.           |
| Niobium*    | Ingot        | 99.9+                    | Kawecki Chemical Company<br>(New York) |
| Vanadium*   | Rod          | 99.6                     | Vanadium Corporation of America        |
| Rhenium     | Sintered rod | 99.9+                    | Chase Brass and Copper                 |
| Osmium      | Powder       | 99.8                     | Fideliton Inc. (Chicago)               |
| Chromium    | Flake        | 99.9                     | Electro Metallurgical Company          |

\* These materials were electron-bombardment melted prior to use.  
Purity indicated is that before melting.

were conventional. A variety of etching reagents was found useful on a trial-and-error basis. These include:

- a. 60 cc glycerine  
20 cc HF  
20 cc HNO<sub>3</sub>  
20 cc H<sub>2</sub>O<sub>2</sub>
  - b. 50 cc HF  
50 cc HNO<sub>3</sub>
  - c. 5 cc H<sub>2</sub>SO<sub>4</sub>  
95 cc H<sub>2</sub>O
- Electrolytic etch, 3 volts DC

Probably the most frequent difficulties encountered was that of distinguishing between cored and two-phase structures. Many of the specimens examined showed strong coring. Sometimes, only by very careful etching procedures can a clear distinction be made. Of course, as a final resort an X-ray powder pattern can resolve the difficulty. A number of alloys contained distributions of insoluble phases which were extraneous to the binary or ternary phase equilibria. These largely derive from interaction between the small amounts of interstitial elements and one of the metallic elements of the system. These had to be distinguished from the development of precipitates by appropriate constitutional phase changes. In general, the condition of the grain boundary was the determining factor in such decisions. The occurrence of banding, apparently twin banding, in the body-centered cubic solid solutions was very commonly encountered. These must not be confused with the Widmanstätten geometry of precipitation of a second phase. A distinction can be easily made because in any coarse precipitation structure the grain boundary will be heavily populated with the new phase. In the banded structure observed, the grain boundaries were invariably free of any suggestion of a second phase.

X-ray diffraction patterns were made on crushed or filed powders using copper K<sub>α</sub> radiation. The powders were sealed in Vycor ampules and annealed at 1000°C for 15 minutes prior to examination. Accurate lattice-parameter measurements were made by using the Nelson-Riley system of extrapolation to eliminate error.

### III. BINARY SYSTEMS

Each of the pertinent binary systems will be discussed individually. Work performed under this research program has been integrated with the published literature but properly identified. In several instances it has been found that the data from ternary systems can be taken to assist more accurate placement of solid solubility boundaries. Such instances are cited.

All compositions are reported in weight percentages. Microstructures are used only to illustrate critical points in phase equilibria. A tabular summary of the binary systems is presented in Table II.

#### 1. Tungsten-Tantalum System

References 1 to 4 show the existence of continuous solid solution between the two components and no intermediate phases.

#### 2. Tungsten-Molybdenum System

References 5 to 8 established a continuous solid solution between components with no intermediate phases.

#### 3. Tungsten-Vanadium System

According to work on a contemporary research program (9), a continuous solid solution exists between the components. No intermediate phases exist.

#### 4. Tungsten-Niobium System

Reference 3 established the existence of a continuous solid solution between components with no intermediate phases.

#### 5. Tungsten-Chromium System

According to references 10 to 12, a continuous solid solution occurs between the components above 1500°C. A solid state immiscibility field exists below this temperature, according to references 11 and 12. The preferred results of reference 12 are graphically illustrated in Figure 1.

#### 6. Tungsten-Rhenium System

The diagram of reference 13 is accepted for the work and is reproduced in Figure 2.

#### 7. Tungsten-Osmium System

Four alloys containing, respectively, 3.1, 7.3, 15.4, and 25.7% Os, were prepared and studied. In the as-cast condition, the 15.4 and 25.7% Os alloys contained, by visual estimate, about 70% and 10% primary dendrites of tungsten solid solution, respectively. As a first approximation this indicates the maximum solid solubility of osmium in tungsten to be about 10%. The 7.3% Os alloy annealed at 1500°C showed precipitation from solid solution in a Widmanstätten pattern indicating a solubility limit at that temperature of about 7% Os. Annealed at 1000°C, the 3.1% Os showed only a single-phase structure indicating the solubility limit at this temperature greater than this and estimated at about 5% Os.



TABLE II  
SUMMARY OF BINARY DIAGRAM DATA

| System | Maximum Solid Solubility                  | Compounds                                  | Remarks       |
|--------|---|--|---------------|
| W-Ta   | Complete solid solubility                 |  |               |
| W-Mo   | Complete solid solubility                 |  |               |
| W-V    | Complete solid solubility                 |  |               |
| W-Nb   | Complete solid solubility                 |  |               |
| W-Cr   | Complete solid solubility<br>above 1500°C |  | See Figure 1  |
| W-Re   | 38% Re                                    | $\sigma$ , 41-65% Re<br>$\chi$ , 72-77% Re | See Figure 2  |
| W-Os   | 10% Os                                    | $\sigma$ , 34-36% Os                       |               |
| Ta-Mo  | Complete solid solubility                 |  |               |
| Ta-V   | Complete solid solubility<br>above 1320°C | TaV <sub>2</sub> , 36% V                   | See Figure 3  |
| Ta-Nb  | Complete solid solubility                 |  |               |
| Ta-Cr  | 10% Cr                                    | TaCr <sub>2</sub> , 37% Cr                 | See Figure 5  |
| Ta-Re  | 51-55% Re                                 | $\sigma$ , 60% Re<br>$\alpha$ , 66-84% Re  | See Figure 6  |
| Ta-Os  | 16% Os                                    | $\sigma$ , 26-37.5% Os                     |               |
| Mo-V   | Complete solid solubility                 |  |               |
| Mo-Nb  | Complete solid solubility                 |  |               |
| Mo-Cr  | Complete solid solubility                 |  |               |
| Mo-Re  | 60% Re                                    | $\sigma$ , 69-81% Re<br>$\chi$ , 83-88% Re | See Figure 9  |
| Mo-Os  | 25% Os                                    |  |               |
| V-Nb   | Complete solid solubility                 |  |               |
| V-Cr   | Complete solid solubility                 |  |               |
| V-Re   | 89% Re                                    | $\sigma$ , 86-92% Re                       |               |
| V-Os   | 50% Os                                    | VOs, 78.9% Os                              |               |
| Nb-Cr  | 25% Cr                                    | NbCr <sub>2</sub> , 52.8% Cr               | See Figure 13 |
| Nb-Re  | 57% Re                                    | $\sigma$ , 65-69% Re<br>$\chi$ , 75-79% Re | See Figure 17 |

TABLE II (Continued)

| System | Maximum Solid Solubility | Compounds                                  | Remarks |
|--------|--------------------------|--|---------|
| Nb-Os  | 32% Os                   | $\beta$ , 110.5% Os<br>$\alpha$ , 72.3% Os |         |
| Cr-Re  | >70% Re                  | 85.3% Re                                   |         |
| Cr-Os  | 75% Os                   |  |         |
| Re-Os  | Complete solubility      |  |         |

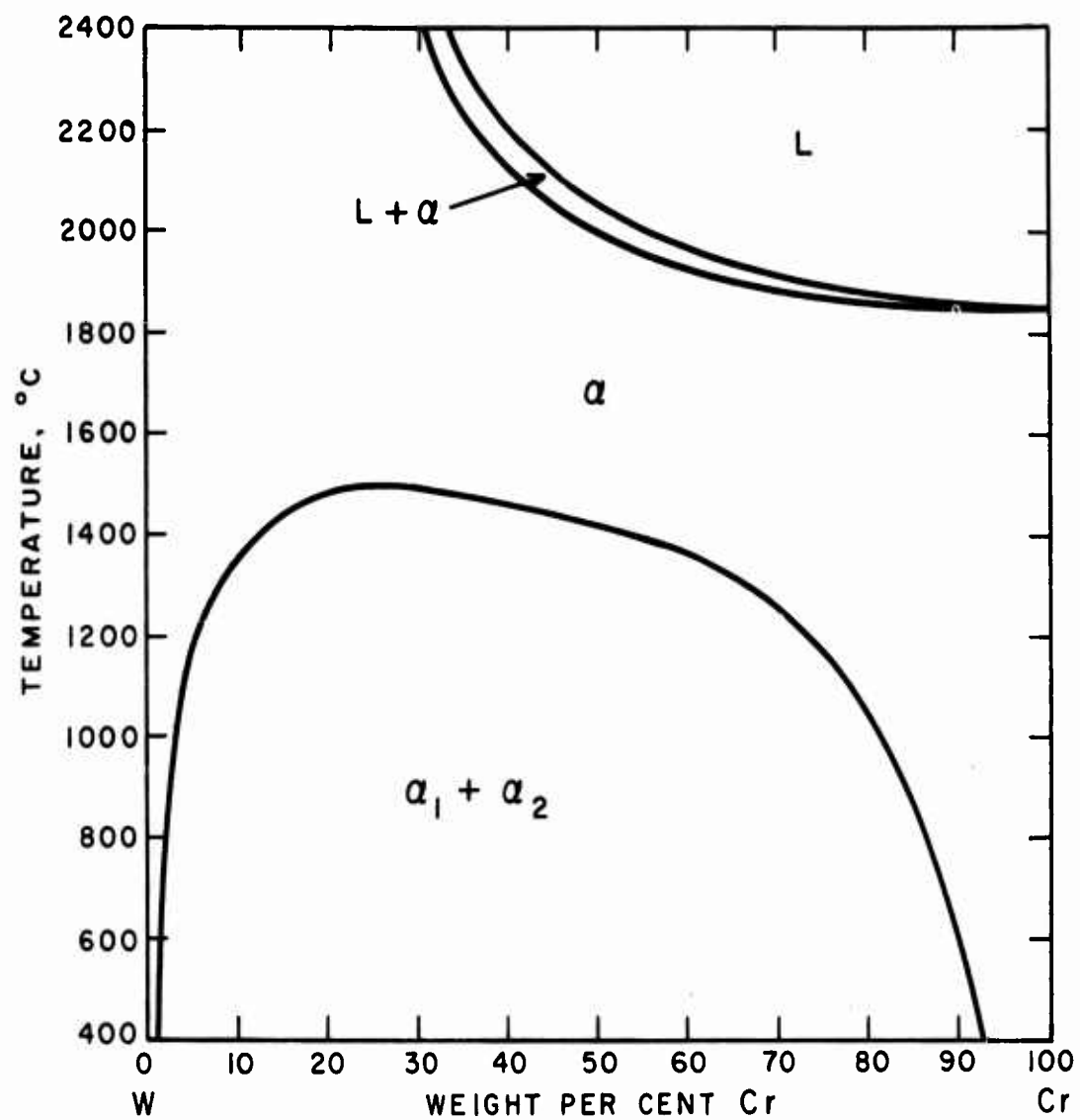


FIG. 1 - THE TUNGSTEN-CHROMIUM SYSTEM  
(Reference 12)

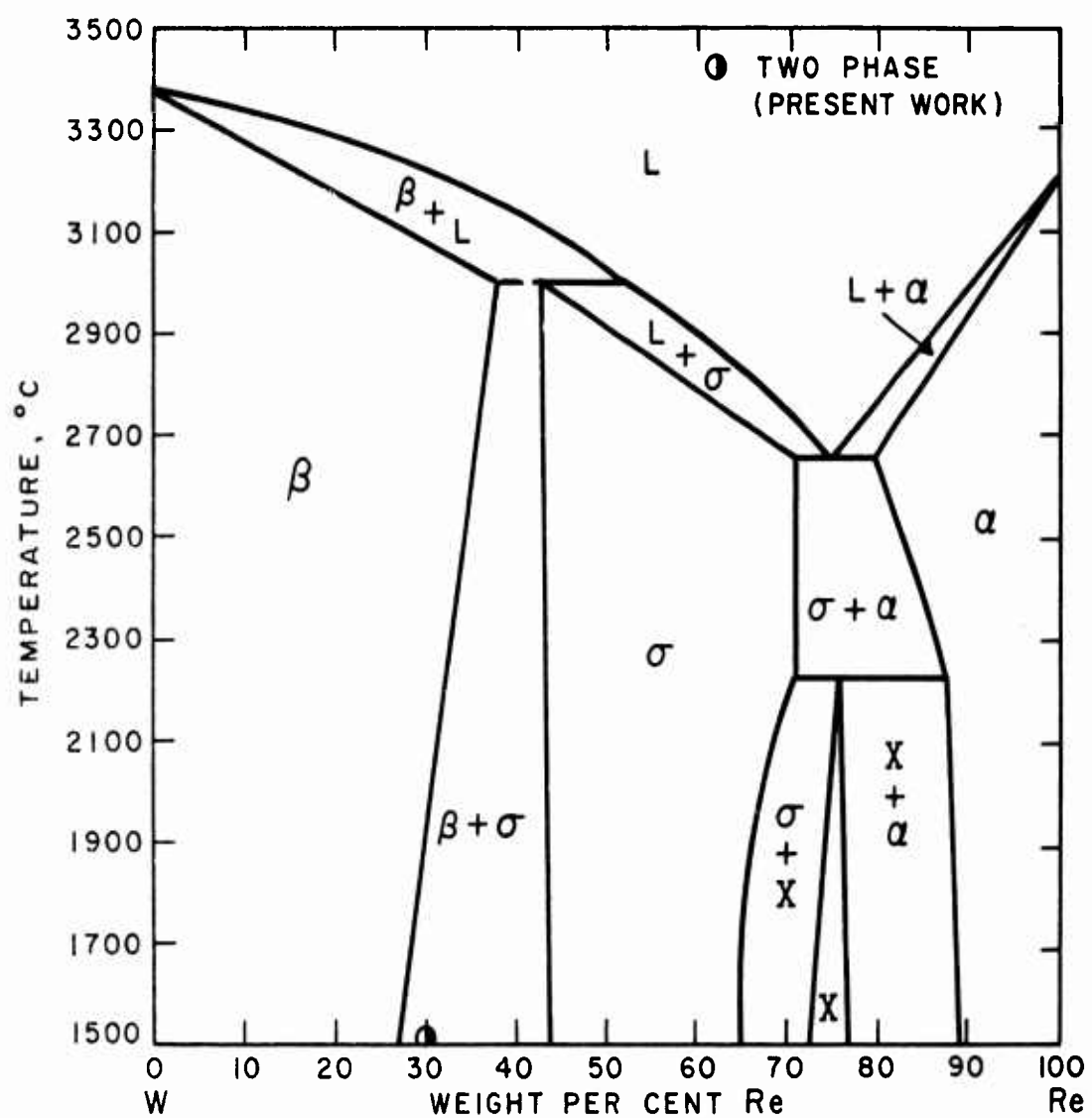


FIG. 2 - THE TUNGSTEN-RHENIUM SYSTEM

(Reference 13)

References 14 to 17 identify a sigma-type intermediate phase in the region 34 to 36% Os. In support of this, the 25.7% Os alloy was two-phase with about 90% of the intermetallic compound.

#### 8. Tantalum-Molybdenum System

References 3, 4, 18 and 19 demonstrated that a continuous solid solution between components occurs and no intermediate phases exist.

#### 9. Tantalum-Vanadium System

According to reference 20, complete miscibility exists above 1320°C. Below this temperature an intermediate phase  $TaV_2$  (36% V) precipitates. The boundaries of the two-phase zones are given in Figure 3. The structure of the  $TaV_2$  phase was shown (21) to be of the Laves family, having a face-centered cubic structure isomorphous with  $MgCu_2$ .

#### 10. Tantalum-Niobium System

Reference 3 showed that complete miscibility exists in this system, with no intermediate phases.

#### 11. Tantalum-Chromium System

One intermediate phase,  $TaCr_2$  (37% Cr) has been identified by references 4, 22, 23, 24. The phase is one of the Laves family, is hexagonal and isomorphic with  $MgZn_2$ , and is thought to have allotropy.

On the chromium-rich side,  $TaCr_2$  and Cr enter into a eutectic reaction at about 1700°C and 66% Cr. Alloys for further elucidation were made at 1.3, 6, 9.4, 15.3, and 20.2% Cr. The last three alloys give clear evidence of a eutectic solidification on the tantalum side of  $TaCr_2$ . The 20.2% Cr alloy, as shown in Figure 4, is almost at the eutectic composition. The solid solubility of chromium in tantalum appears to be 5% at 1500°C and is estimated at 4% at 1000°C. An approximation of the tantalum-chromium diagram is given in Figure 5.

#### 12. Tantalum-Rhenium System

According to reference 25, the solid solubility of rhenium in tantalum is 49-55% and two intermediate phases exist--a sigma-type phase at about 60% Re and an alpha manganese-type phase at 66-84% Re. Reference 26 essentially confirms these points but sets the solid solubility limit at slightly less than 50% Re. The diagram is given in Figure 6.

In the present work alloys containing 40.7, 51, 60, and 70% Re were prepared. The first two alloys were within the terminal Ta-base solid solution range as verified by the continuity of lattice parameters shown in Figure 7. The 70% Re alloy was single-phase in accordance with the above-mentioned existence of intermediate phases. The solid solubility limit is assumed to be between 51 and 55% Re.

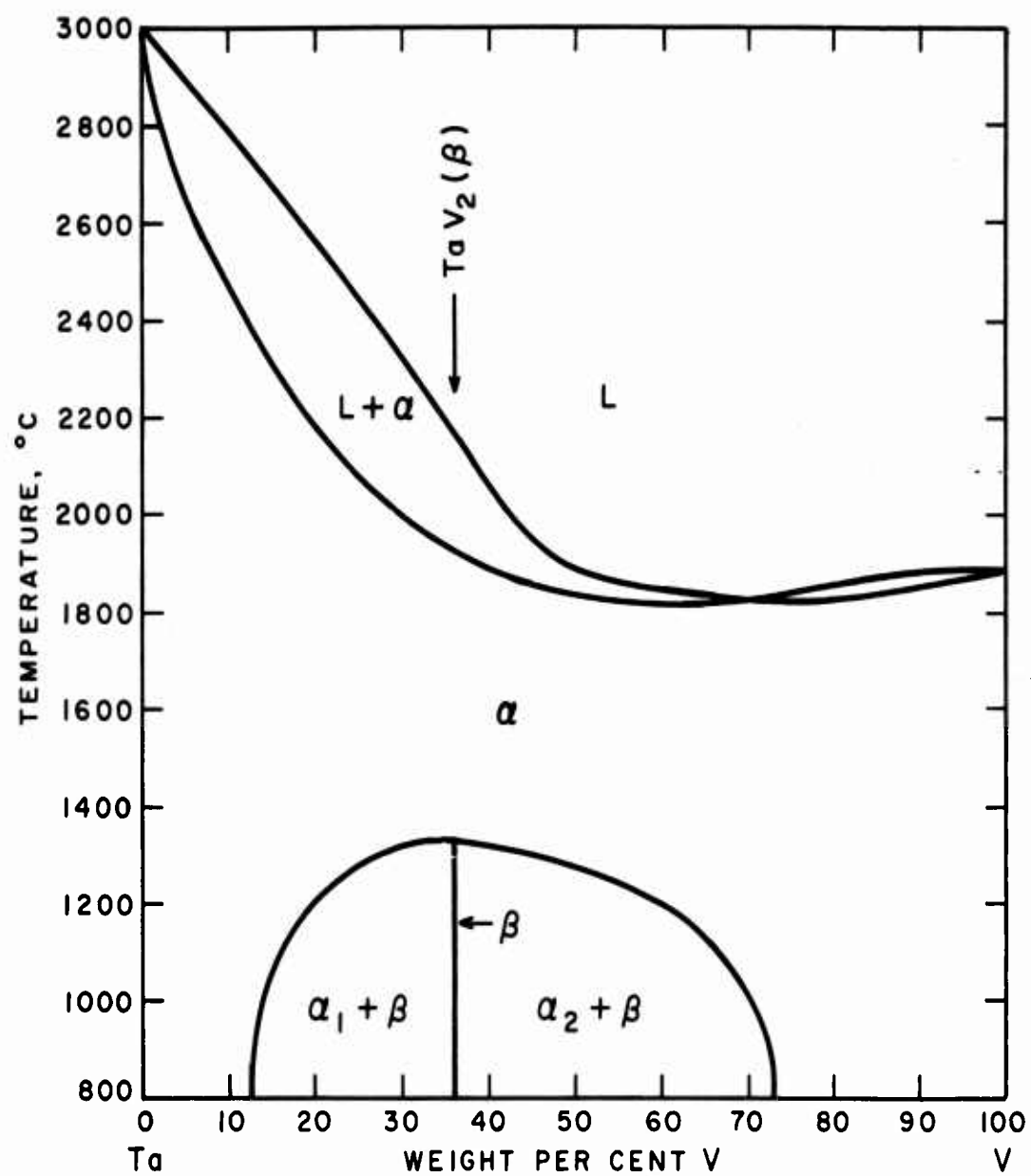
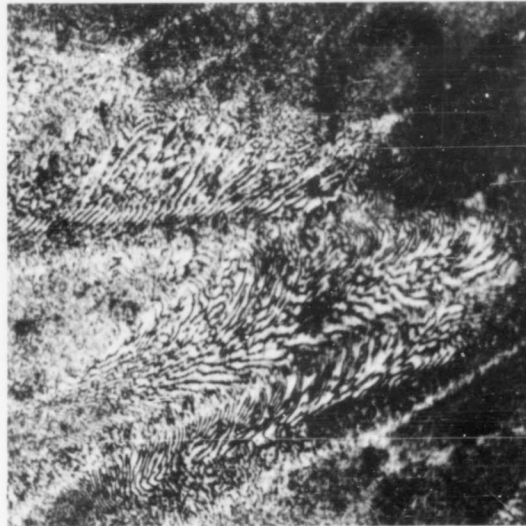


FIG. 3 - THE TANTALUM-VANADIUM SYSTEM

(Reference 20)



Neg. No. 18652

X 500

Fig. 4

As-Cast Structure of Ta-20.2% Cr Alloy  
Showing an Almost All-Eutectic Structure  
of Ta and TaCr<sub>2</sub>. Electrolytically Etched  
with 5% H<sub>2</sub>SO<sub>4</sub>.

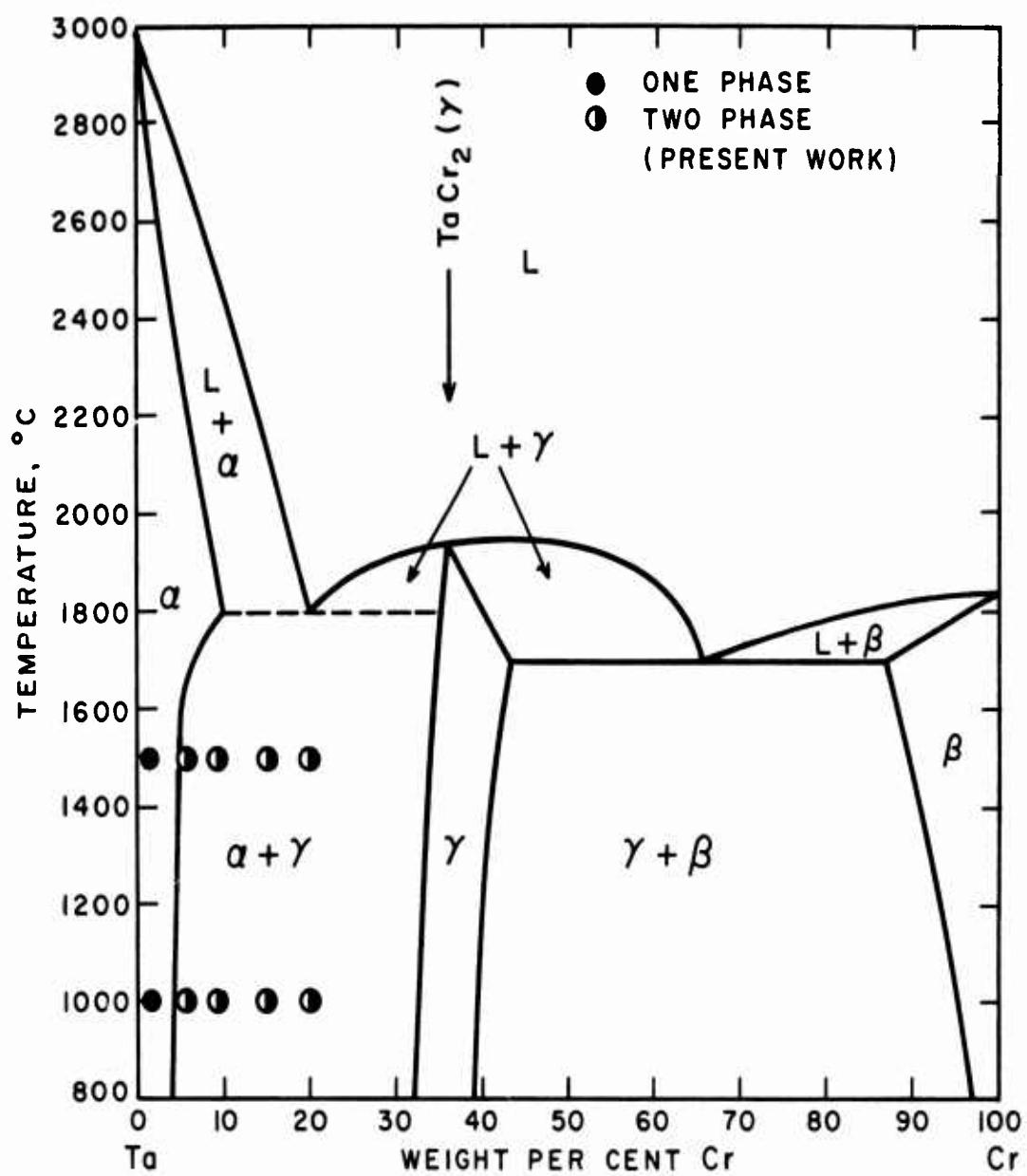


FIG. 5 - THE TANTALUM-CHROMIUM SYSTEM

(References 4, 22, 23, 24 and present work)



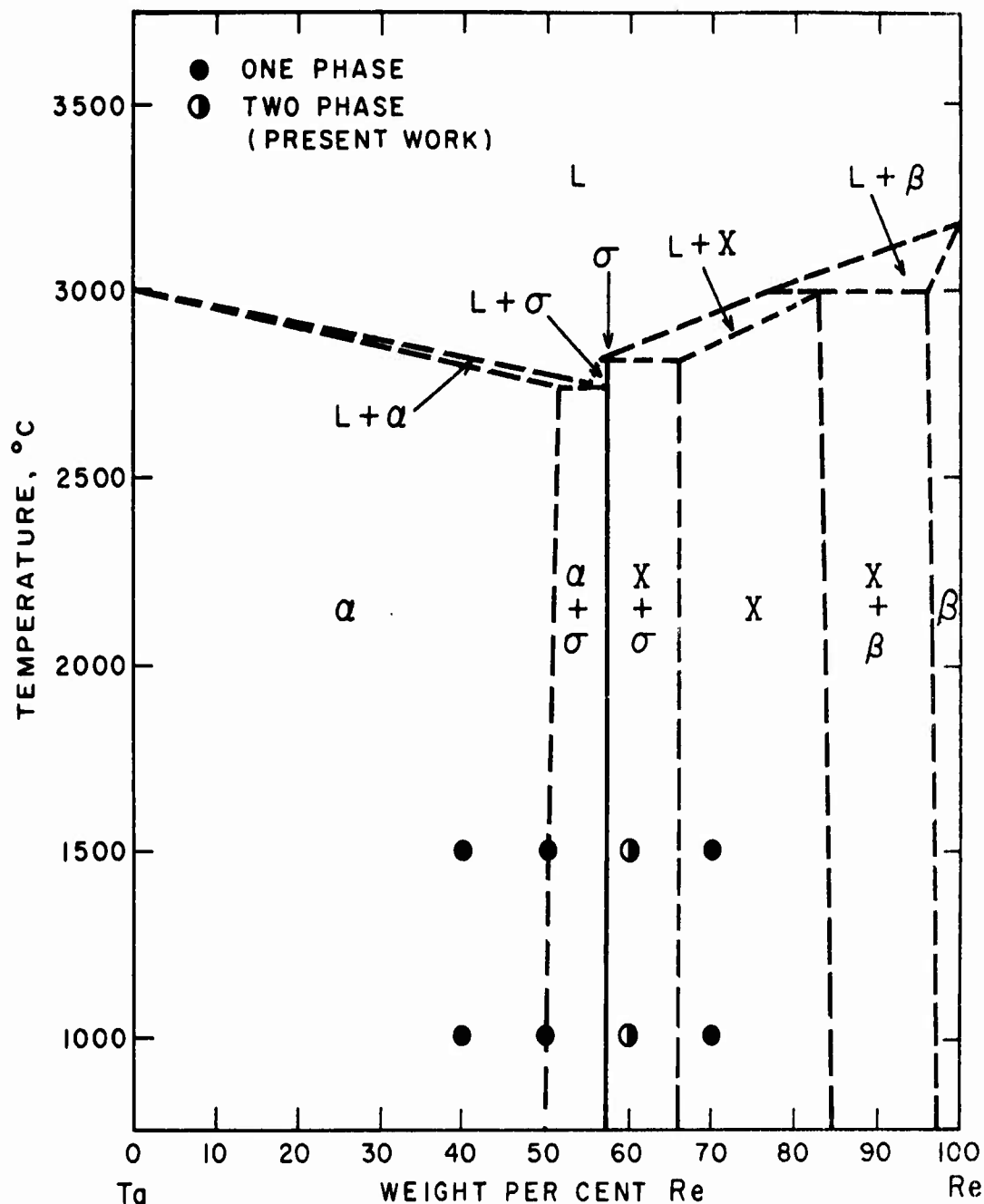


FIG. 6 - THE TANTALUM-RHENIUM SYSTEM \*

(Reference 26 and present work)

\*The binary diagram shown (from reference 26) is a tentative diagram based on the preliminary data that was available at the time the author prepared this text. It has since been revised by Wulff based upon later experimental data and will be published in WADD Technical Report 60-132 "Constitution Diagrams of Refractory Metals". The revised diagram shows that the  $\sigma$  phase is not stable below 2500°C. This revision affects the tentative phase relationships shown in figures 25, 46, 50, 55, 58 and 59 which involve the Ta-Re system.

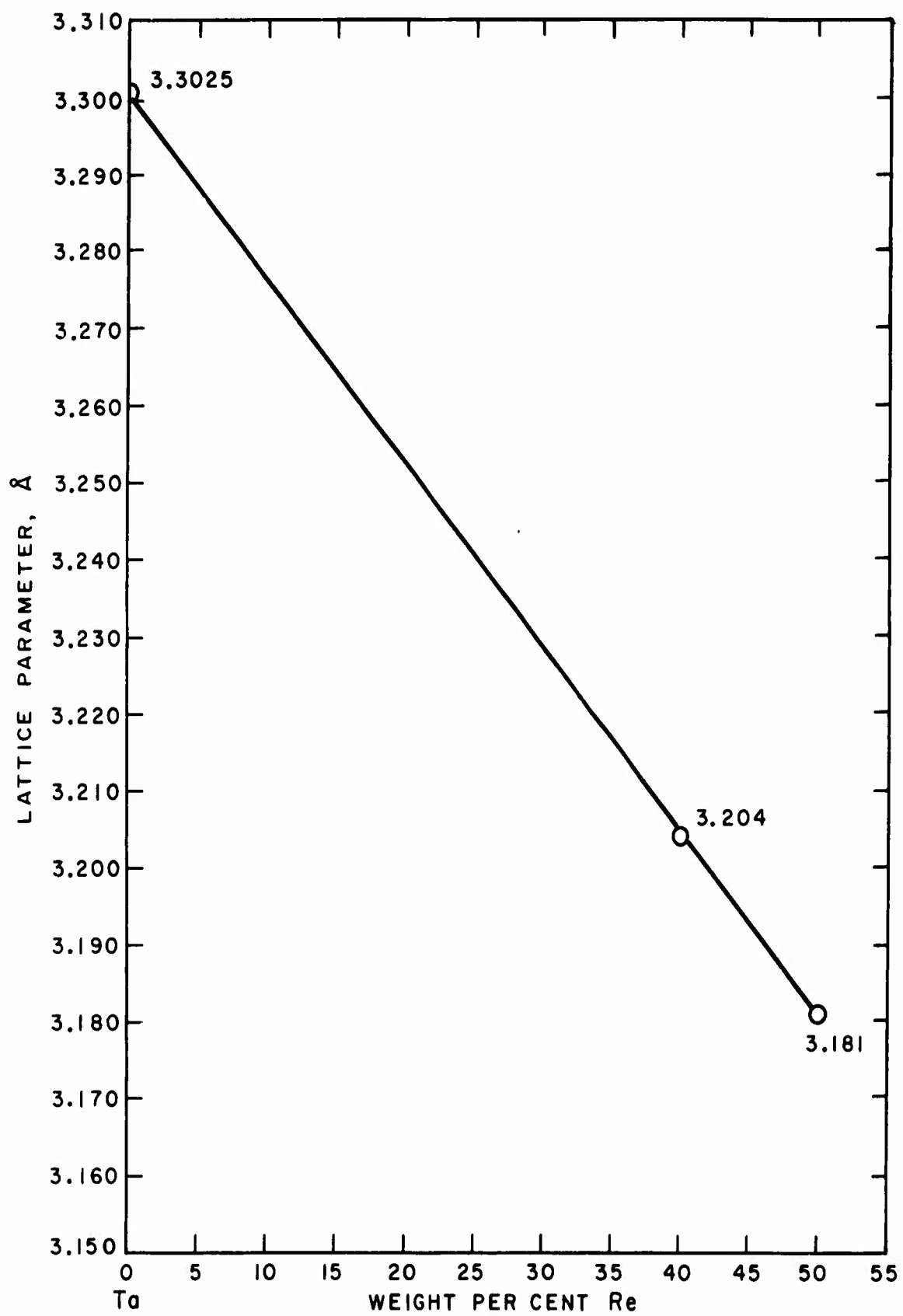


FIG. 7 - LATTICE PARAMETERS OF TANTALUM-RHENIUM  
SOLID SOLUTION

### 13. Tantalum-Osmium System

Reference 14 reports the existence of a sigma-type intermediate phase at 26-37.5% Os. In the present work alloys containing 10.4, 15, 20.8, 31.1% Os, respectively, were melted. The 15% Os alloy was shown to exist already in the two-phase field. The 10.4% Os alloy was single-phase at all temperatures. Limited lattice-parameter data in Figure 8 indicate that the solid solubility limit is about 16% Os. Data from the ternary systems Ta-Nb-Os and Ta-Os-Re reinforce this conclusion. The 20.8% Os alloy has a two-phase structure, and the 31.1% Os alloy is single-phase.

### 14. Molybdenum-Vanadium System

References 27 and 28 indicate complete miscibility of the two components. There has been the suggestion of an intermediate phase forming in the solid state at low temperatures (28). In the present work alloys containing 21, 35, 43.9, and 51.6% V, respectively, were melted. Only single-phase structures were encountered.

### 15. Molybdenum-Niobium System

References 3 and 4 agree that complete miscibility in the solid state occurs in this system.

### 16. Molybdenum-Chromium System

References 29 showed complete solid state miscibility and no intermediate phases.

### 17. Molybdenum-Rhenium System

The phase diagram given by reference 13 is accepted and reproduced in Figure 9.

### 18. Molybdenum-Osmium System

References 25 and 30 indicate a low solubility of osmium in molybdenum. Two intermediate phases have been observed at 39.8% Os and 54.3% Os, respectively. The first intermediate phase was reported to have a  $\beta$ -tungsten type structure and, from other compounds with this structure, probably indicates a stoichiometry of  $\text{Mo}_3\text{Os}$ .

In the present work, alloys containing 9.4, 18, 30, and 40% Os were melted. In the as-cast state, the 30 and 40% alloys were two-phase structures which seems to deny the existence of  $\text{Mo}_3\text{Os}$ . The 9.4 and 18% Os alloys were single-phase, as cast. On annealing at 1500°C, the 18% Os alloy showed the Widmanstätten character of a precipitant from the solid state (see Figure 10). The maximum solid solubility is therefore in excess of 18% Os (probably about 25% Os), and solubility at 1500°C as well as at 1000°C is less than 18% Os (by visual estimate at about 15% Os).

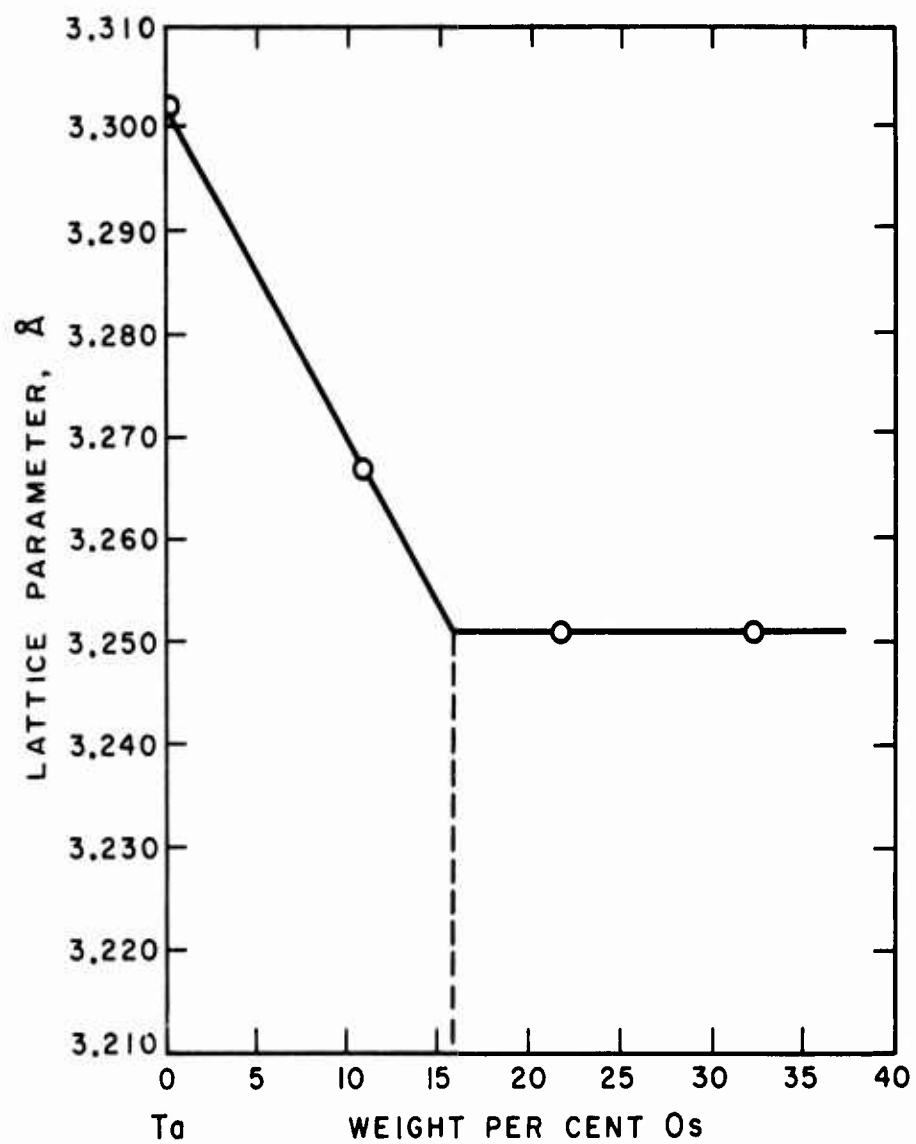


FIG. 8 - LATTICE PARAMETERS OF THE TANTALUM-RICH  
SOLID SOLUTION IN THE TANTALUM-OSMIUM SYSTEM

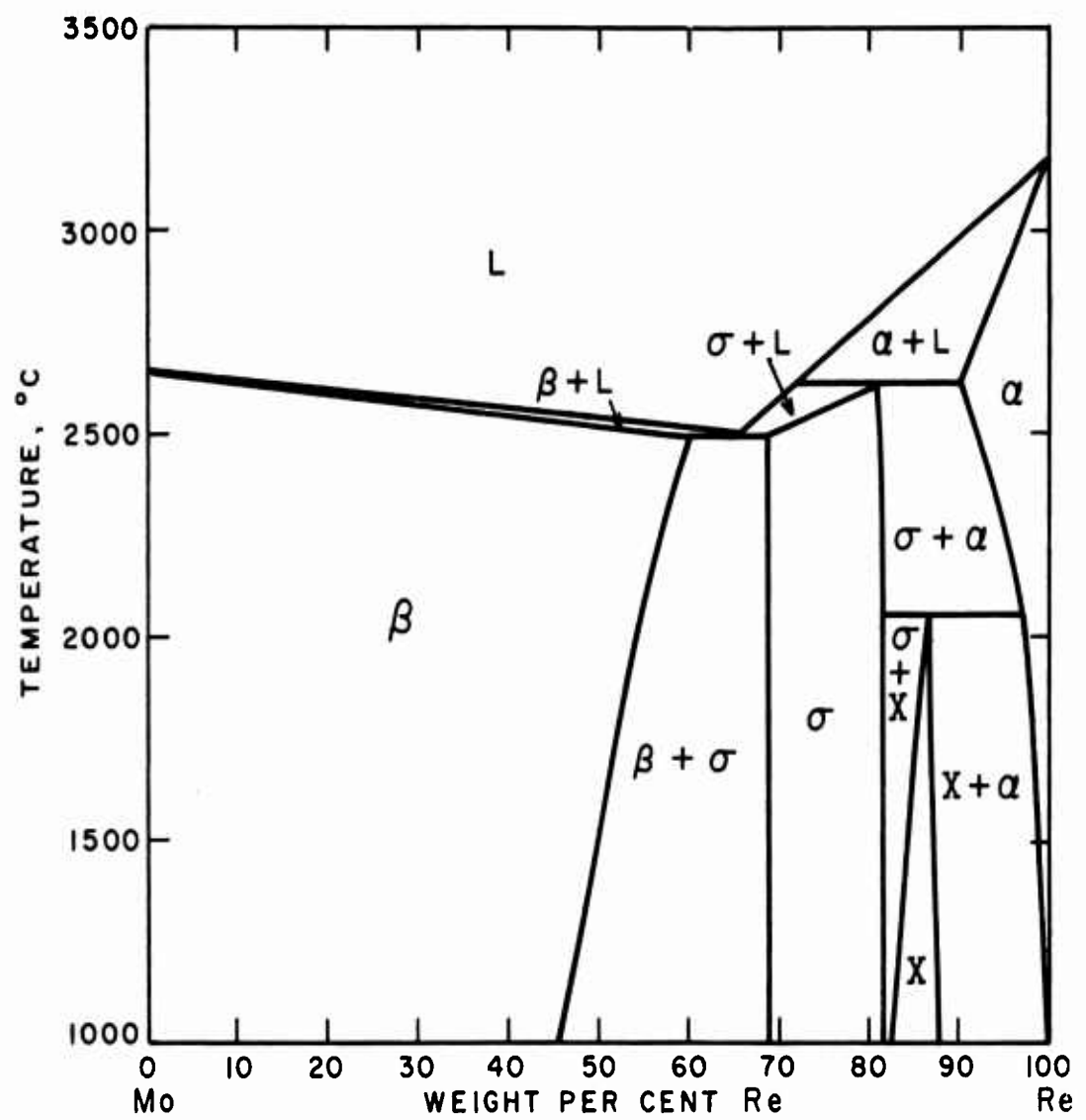
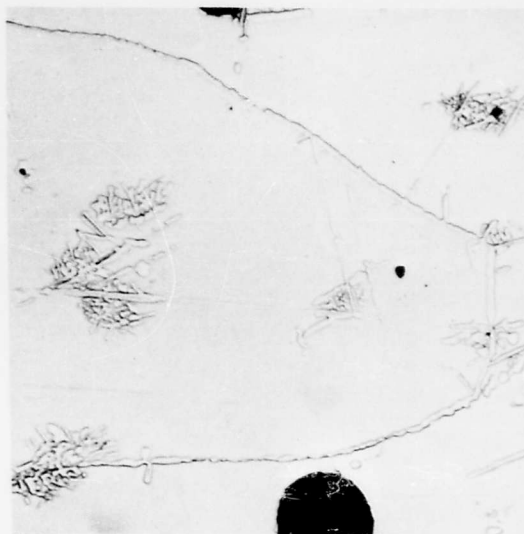


FIG. 9 - THE MOLYBDENUM-RHENIUM SYSTEM  
(Reference 13)



Neg. No. 18655

X 500

Fig. 10

Microstructure of a Mo-18% Os Alloy  
Annealed at 1500°C for 3/4 hr Showing  
Precipitation from Solid Solution.  
Electrolytically Etched with 5%  $\text{H}_2\text{SO}_4$ .

#### 19. Vanadium-Niobium System

The work of reference 31, indicating that complete solid state miscibility exists, is accepted.

#### 20. Vanadium-Chromium System

Reference 20 shows that complete solid state miscibility exists.

#### 21. Vanadium-Rhenium System

From the work in a contemporary program (9), the probably phase diagram is reproduced as Figure 11.

#### 22. Vanadium-Osmium System

Reference 15 indicates the existence of a VO<sub>3</sub> (78.9% Os) phase with a CsCl type structure. In the present work, alloys containing 16.4, 39.1, 50, and 60% Os, respectively, were melted.

The 16.4, 39.1, and 50% Os alloys showed a continuous solid solution range. At 1500°C the 60% Os alloy develops a small amount of precipitate at the grain boundaries, as shown in Figure 12. The solid solubility of osmium in vanadium must occur at just less than 60% Os. The 50% Os alloy annealed at 1000°C shows evidence of a very fine precipitate at sub-grain boundaries, indicating a solubility of 50% at 1000°C.

#### 23. Niobium-Chromium System

References 32, 33, and 34 confirm the existence of a NbCr<sub>2</sub> (58.8% Cr) phase, belonging to the Laves family and having a cubic structure of the MgCu<sub>2</sub> type. The diagram of reference 33 shows two eutectic reactions on either side of the intermediate phase at 35 and 69% Cr and about 1710° and 1660°C, respectively. The NbCr<sub>2</sub> phase is purported to melt at 1750°C. The maximum solid solubility of Cr in Nb is estimated at about 25% Cr and of Nb in Cr at about 5% Nb.

In the present work alloys containing 1.9, 5.4, 8.7, 10.5, 20 and 85% Cr were melted. The 8.7 Cr alloy solidified as a heavily cored single phase. On annealing at 1500°C, a trace of precipitate was observed at grain boundaries. A 1000°C anneal produced a large volume of precipitate. The maximum solid solubility is therefore greater than 8.7% Cr; the solubility at 1500°C is about 8% Cr and at 1000°C less than 8% but greater than 5.4% Cr, probably about 7% Cr. The cast structures of the 20% and 85% Cr alloys verified the existence of eutectic solidification reactions on either side of NbCr<sub>2</sub>. The Eremenko diagram modified by the present results is shown in Figure 13. The microstructures of the 8.7% Cr alloy are illustrated in Figures 14 (a) and (b).

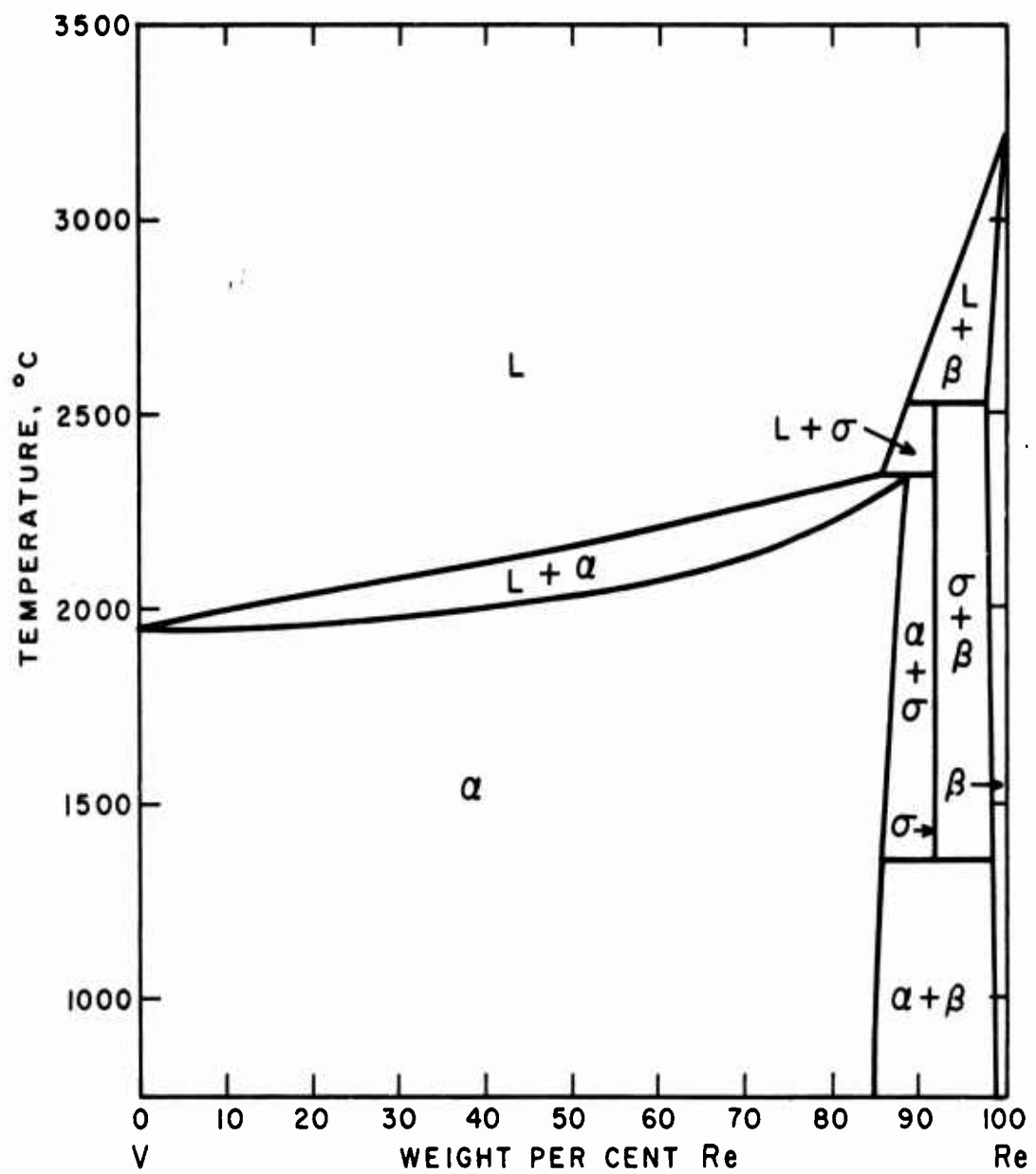
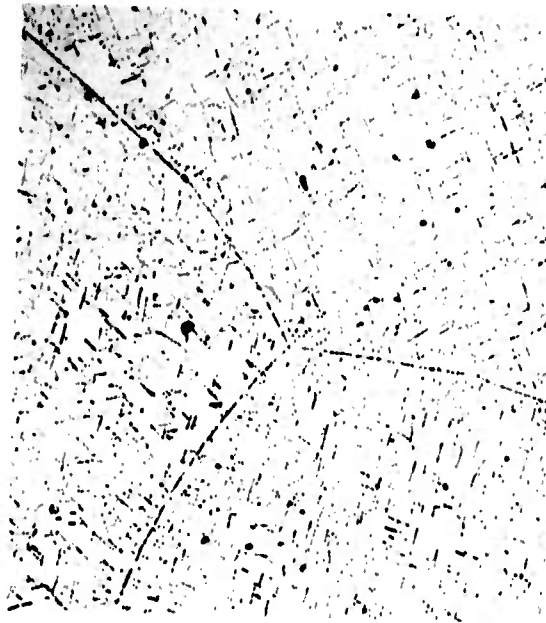


FIG. 11 - THE VANADIUM-RHENIUM SYSTEM

(Reference 9)





Neg. No. 18656

X 500

Fig. 12

Microstructure of a V-60% Os Alloy Showing that this Composition is Just at the Solid Solubility Limit of the V-Rich Solid Solution. Etched with 60cc Glycerine, 20cc  $\text{HNO}_3$ , and 20cc  $\text{H}_2\text{O}_2$ .

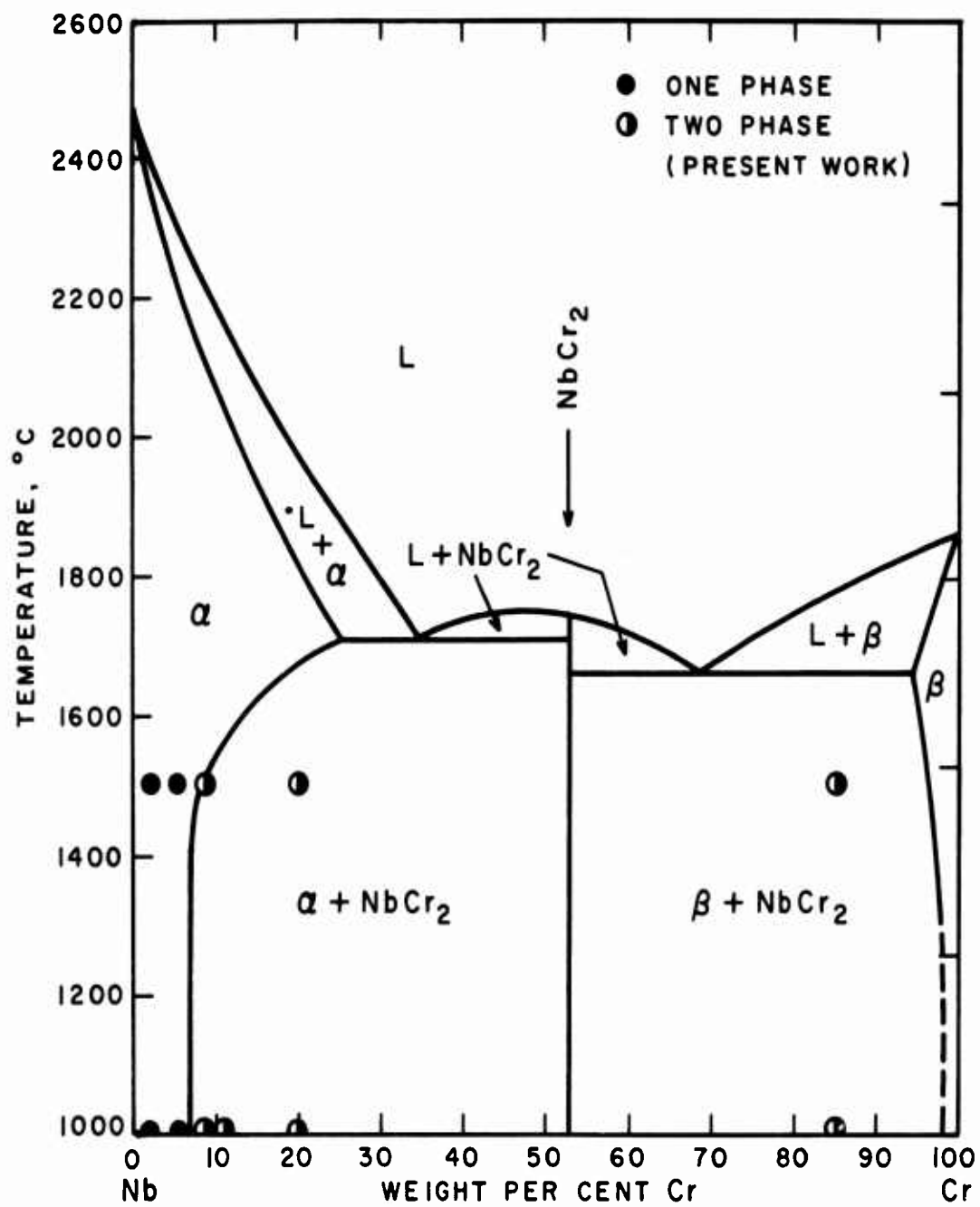
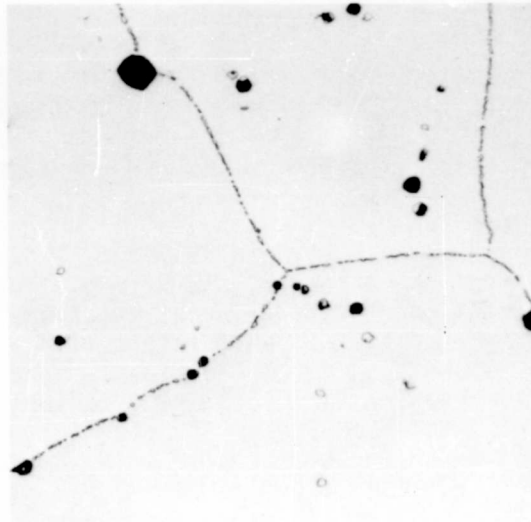


FIG. 13 - THE NIOBIUM-CHROMIUM SYSTEM

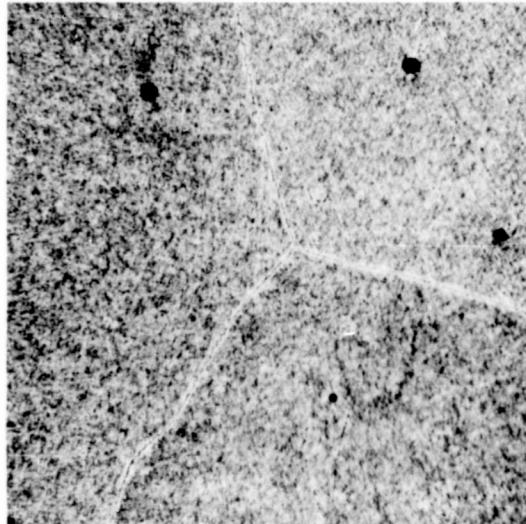
(Reference 33 and present work)



Neg. No. 18653

(a)

X 500



Neg. No. 18657

(b)

X 500

Fig. 14

Microstructures of a Nb-8.7% Cr Alloy  
 Annealed (a) at 1500°C and (b) at 1000°C.  
 Etched with 60cc Glycerine, 20cc HF,  
 20cc  $\text{HNO}_3$  and 20cc  $\text{H}_2\text{O}_2$ .

#### 24. Niobium-Rhenium System

Reference 35 identifies two intermediate phases: one at about 68% Re with a sigma-phase type structure which occurs only below 1000°C and another at 75-88.5% Re and having an alpha-manganese type structure. The maximum solid solubility of rhenium in niobium was reported as 57%.

In the present work, alloys containing 46, 57, 70 and 80% Re were melted. In the as-cast state, the 46 and 57% Re alloys appeared to be single-phase. On annealing at 1500°C, the 57% Re alloy develops a precipitate at the grain boundaries as shown in Figure 15. The solid solubility at this temperature is therefore about 56% Re. The 70% Re alloy in the as-cast state showed the three-phase configuration of an incomplete peritectic reaction (Figure 16.) This indicates that the second intermediate phase is formed by such a reaction. The 80% Re alloy apparently solidified as a single phase. However a precipitate developed on annealing at 1000° and 1500°C. A probably diagram for the Nb-Re system is shown in Figure 17. The temperature of the peritectic reaction is unknown.

#### 25. Niobium-Osmium System

According to references 36 and 37, two intermediate phases exist at 40.5% Os and 72.3% Os, respectively. The former has a  $\beta$ -tungsten type structure and therefore probably has the stoichiometry  $\text{Nb}_3\text{Os}$ . The latter has an  $\alpha$ -Mn type structure.

In the present work, alloys containing 9.7, 18.5, 30, and 40% Os were melted.

The 30% Os alloy proved to be single-phase at 1500°C, whereas the 40% Os alloy had an appreciable amount of second phase. The solid solubility is therefore estimated at about 32% Os, judging from the amount of second phase in the 40% alloy. At 1000°C the 30% Os alloy developed a precipitant of a second phase illustrated in Figure 18. The solubility of osmium in niobium at 1000°C is estimated to be ~28%.

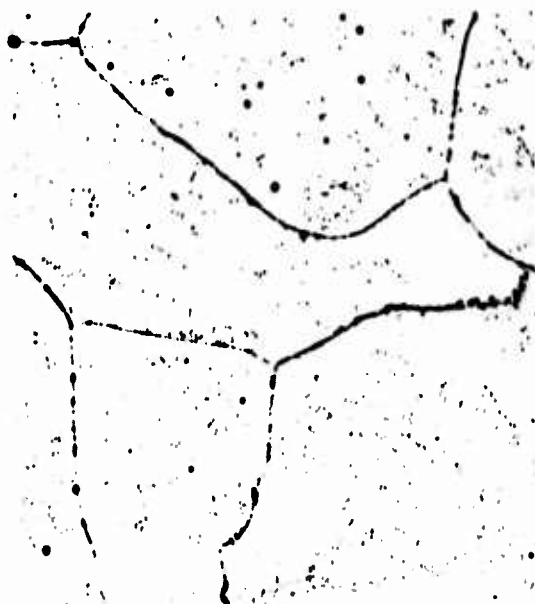
#### 26. Chromium-Rhenium System

References 25, 38, and 39 identify only one intermediate phase at 85.3% Re. Of the two solid solutions on either side, the limits were given as <6.5% Cr in Re and <71% Re in Cr.

Alloys containing 9.5, 19.5, 29, 46, and 68% Re were melted. All of these alloys proved to be single-phase. The alloys melted for the W-Cr-Re system also showed that the solubility limit is high and probably >70% Re.

#### 27. Chromium-Osmium System

Reference 16 reports two intermediate phases:  $\text{OsCr}_3$ , having a cubic  $\beta$ -tungsten type structure, and an  $\text{OsCr}_2$  phase. The maximum solid solubility



Neg. No. 18654

X 500

Fig. 15

Microstructure of a Nb-57% Re Alloy Annealed at 1500°C for 45 Minutes Showing Precipitation from Solid Solution. Electrolytically Etched with 5%  $H_2SO_4$ .



Neg. No. 19108

X 250

Fig. 16

Microstructure of a Nb-70% Re Alloy Showing an Incompleted Peritectic Reaction. The Constituents are Probably Primary Dendrites of Nb Enveloped by the  $\alpha$ -Mn Type Intermediate Phase in a Matrix of What Was Originally Melt. Electrolytically Etched with 5%  $H_2SO_4$ .

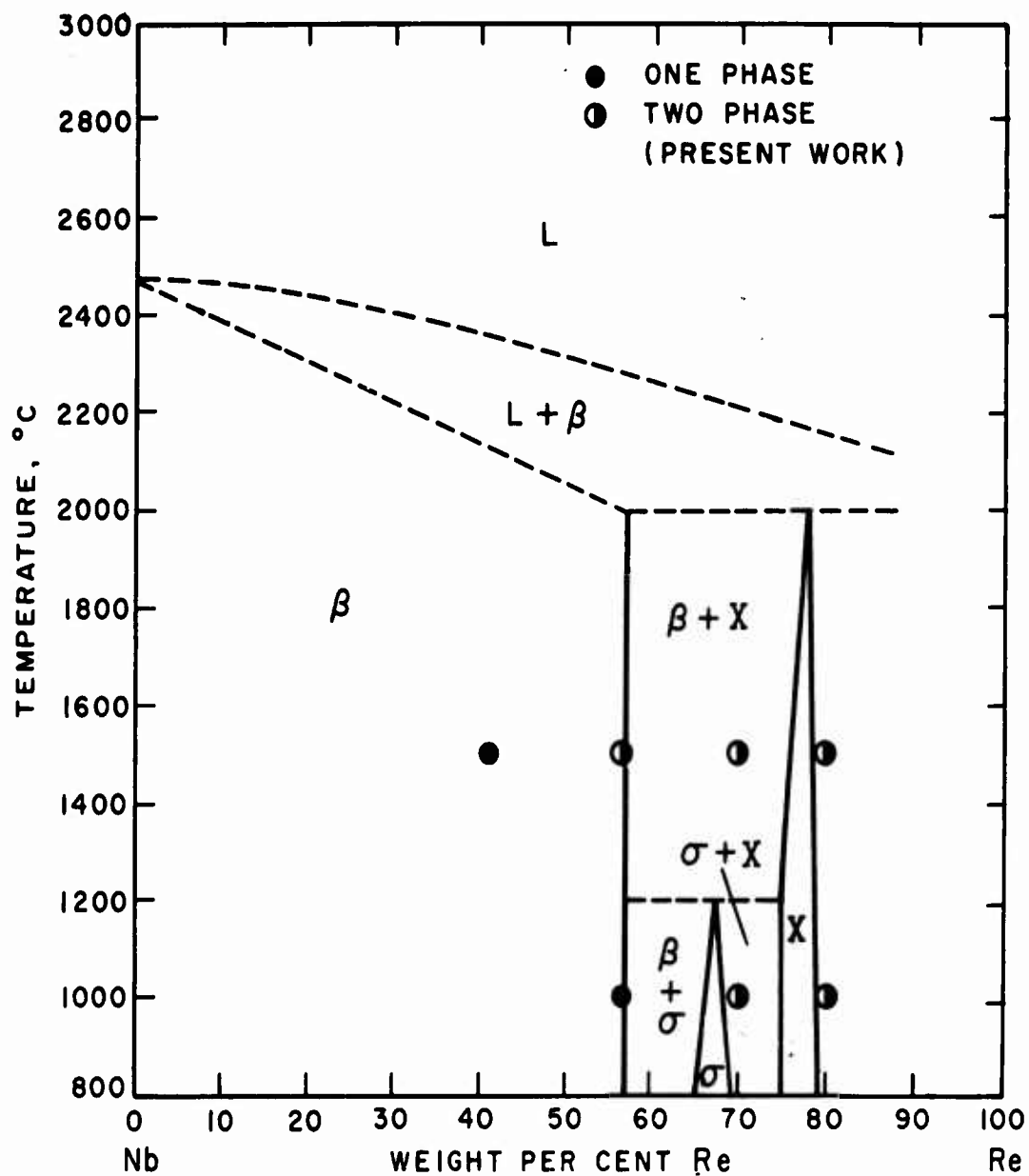


FIG. 17 - THE NIOBIUM-RHENIUM SYSTEM

(Reference 35 and present work)



Neg. No. 18661

X 500

Fig. 18

Microstructure of a Nb-30% Os Alloy Annealed at 1000°C for 100 hours Showing Precipitation of a Second Phase. Electrolytically Etched with 5%  $H_2SO_4$ .

of chromium is osmium was estimated at about 27.8% and of osmium in chromium at about 28%. The  $\text{OsCr}_3$  was regarded as the product of decomposition of  $\text{OsCr}_2$  on cooling.

Alloys containing 16, 29, 35, 40, 50, 60, 66.5% Os were found to be single-phase. An 84% Os alloy was clearly two-phase in the as-cast condition as well as after annealing at 1500° and 1000°C. Accordingly, the solubility of osmium in chromium must be about 75% and the presence of  $\text{OsCr}_2$  and  $\text{OsCr}_3$  must be refuted.

#### 28. Rhenium-Osmium System

Reference 40 indicates complete miscibility between the two components.

### IV. TERNARY SYSTEMS<sup>+</sup>

#### 1. W-Ta-Mo System

|                    |       |   |        |   |        |
|--------------------|-------|---|--------|---|--------|
| Alloys containing: | 22% W | - | 49% Ta | - | 29% Mo |
|                    | 45% W | - | 44% Ta | - | 11% Mo |
|                    | 25% W | - | 49% Ta | - | 26% Mo |

demonstrated complete miscibility of the three components. Based on lattice parameters from these alloys and from binary alloys, an approximate parameter surface is shown in Figure 19.

#### 2. W-Ta-Nb System

|                    |       |   |          |   |          |
|--------------------|-------|---|----------|---|----------|
| Alloys containing: | 50% W | - | 25% Ta   | - | 25% Nb   |
|                    | 45% W | - | 44% Ta   | - | 11% Nb   |
|                    | 25% W | - | 49.5% Ta | - | 25.5% Nb |

demonstrated that the three components are completely miscible. Lattice parameters from these alloys and from binary alloys permitted the construction of the approximate surface shown in Figure 20.

#### 3. W-Ta-V System

Three alloys were prepared in this system:

|  |       |   |        |   |       |
|--|-------|---|--------|---|-------|
|  | 40% W | - | 35% Ta | - | 25% V |
|  | 30% W | - | 40% Ta | - | 30% V |
|  | 20% W | - | 45% Ta | - | 35% V |

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<sup>+</sup> All percentages discussed are on a weight basis.



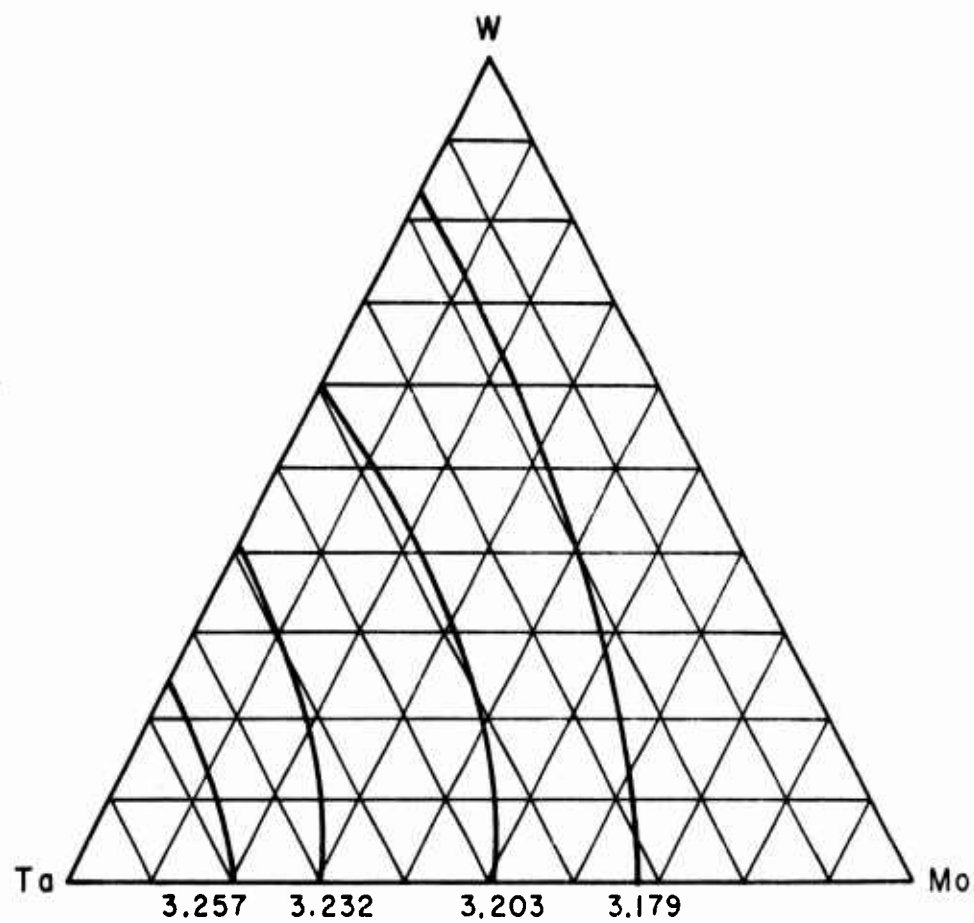


FIG. 19 - APPROXIMATE PARAMETRIC SURFACE  
OF THE W-Ta-Mo SYSTEM ( $\text{\AA}$ )

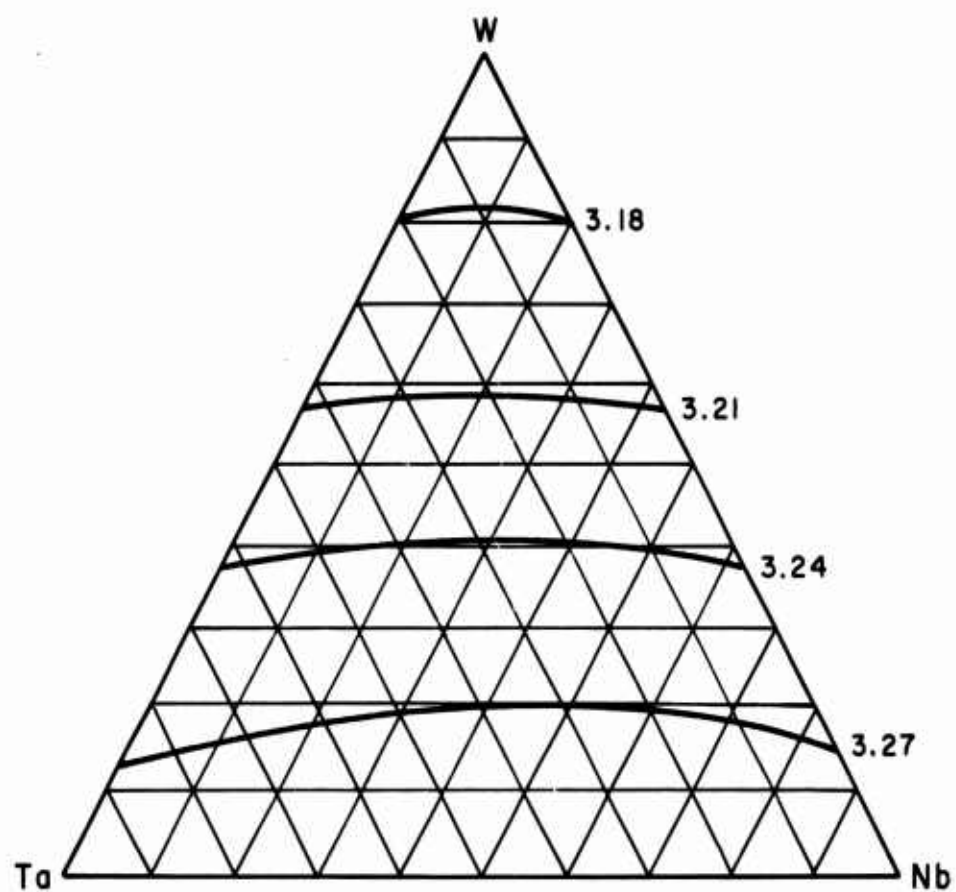


FIG. 20 - APPROXIMATE PARAMETRIC SURFACE  
OF THE W-Ta-Nb SYSTEM ( $\text{\AA}$ )

Complete solid miscibility was confirmed at 1500°C; phase relationships at 1000°C are shown in Figure 21. The fact that the 20% W-45% Ta-35% V alloy had a very small amount of second phase after being annealed at 1000°C indicates the proximity of this composition to the two-phase boundary.

#### 4. W-Ta-Cr System

The following compositions were arc melted:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 20% W | - | 15% Ta | - | 65% Cr |
| 30% W | - | 55% Ta | - | 15% Cr |
| 40% W | - | 30% Ta | - | 30% Cr |
| 50% W | - | 10% Ta | - | 40% Cr |
| 60% W | - | 20% Ta | - | 20% Cr |

All of these alloys contained various proportions of primary dendrites and eutectic characteristic of the Ta-Cr system in the as-cast state. They were likewise two-phase at 1500°C (solid solution + TaCr<sub>2</sub>). At 1000°C, several three-phase structures were observed. Isothermal sections for these two temperatures are presented as Figures 22 and 23.

#### 5. W-Ta-Os System

The following alloys were melted:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 48% W | - | 47% Ta | - | 5% Os  |
| 45% W | - | 44% Ta | - | 11% Os |
| 40% W | - | 39% Ta | - | 21% Os |
| 35% W | - | 34% Ta | - | 31% Os |

The metallographic results are interpreted to give the isothermal section in Figure 24, which is valid both for 1500° and 1000°C, with the single exception of a reduced solid solubility of W in Os to about 5% at 1000°C. The  $\sigma$  phases in the W-Os and Ta-Os systems appear to be completely miscible.

#### 6. W-Ta-Re System

The following alloys were melted:

|       |   |          |   |          |
|-------|---|----------|---|----------|
| 25% W | - | 15% Ta   | - | 60% Re   |
| 25% W | - | 24.5% Ta | - | 50.5% Re |
| 25% W | - | 34.5% Ta | - | 40.5% Re |

In addition to these, two data points were taken from reference 26 to give the composite isothermal section in Figure 25. It would appear that the sigma-type phases of the two binary systems W-Re and Ta-Re are completely miscible. The 25% W-15% Ta alloy in the as-cast state gave evidence of a peritectic mode of solidification. This is illustrated in Figure 26.

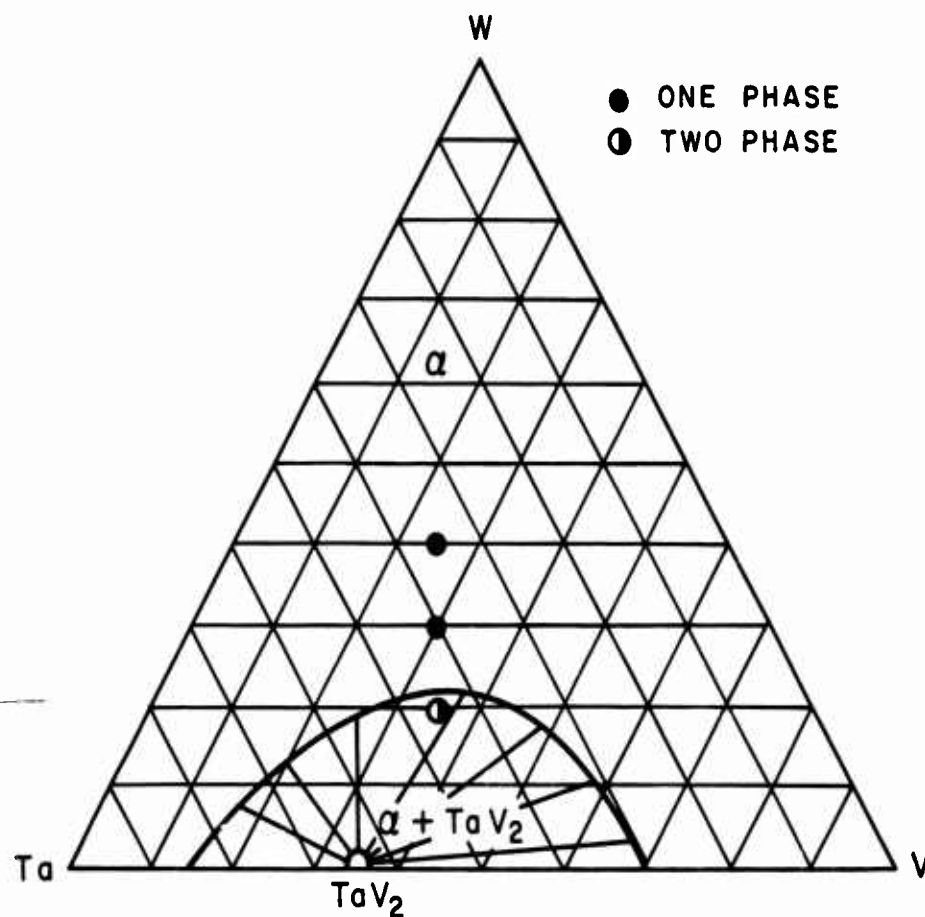


FIG. 21 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Ta-V SYSTEM AT 1000°C

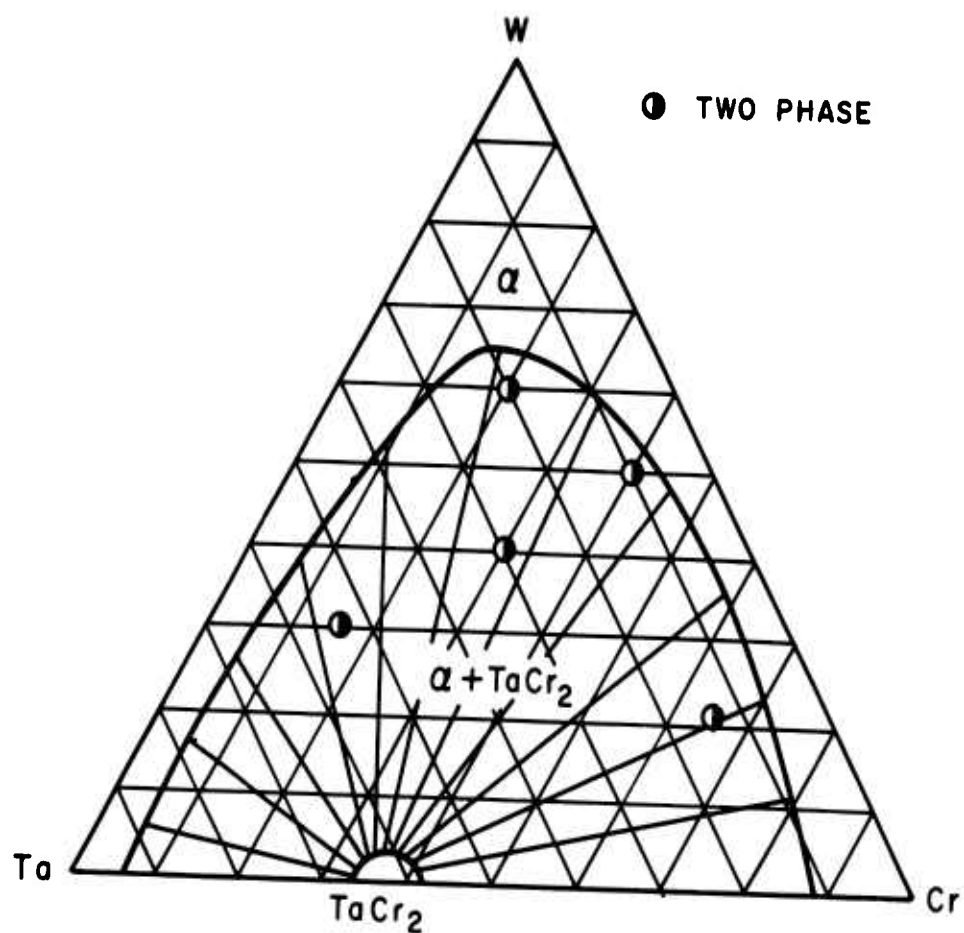


FIG. 22 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Ta-Cr SYSTEM AT 1500 °C.

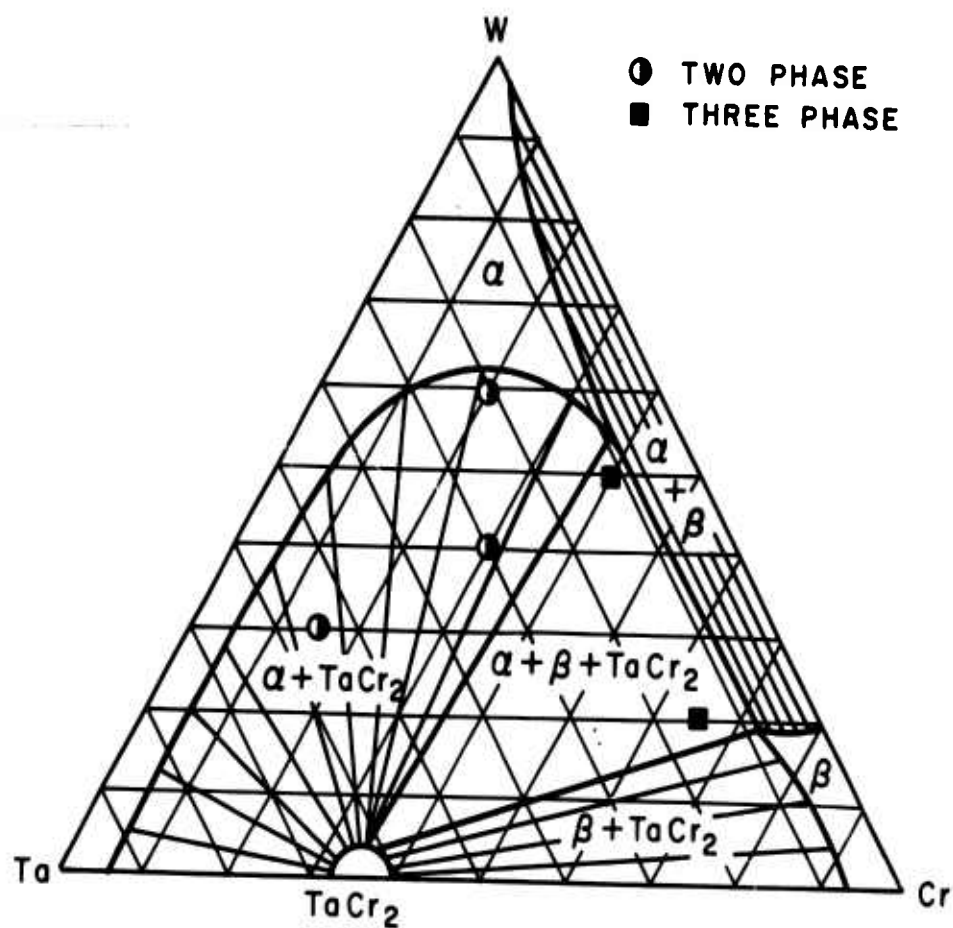


FIG. 23 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Ta-Cr SYSTEM AT 1000°C.

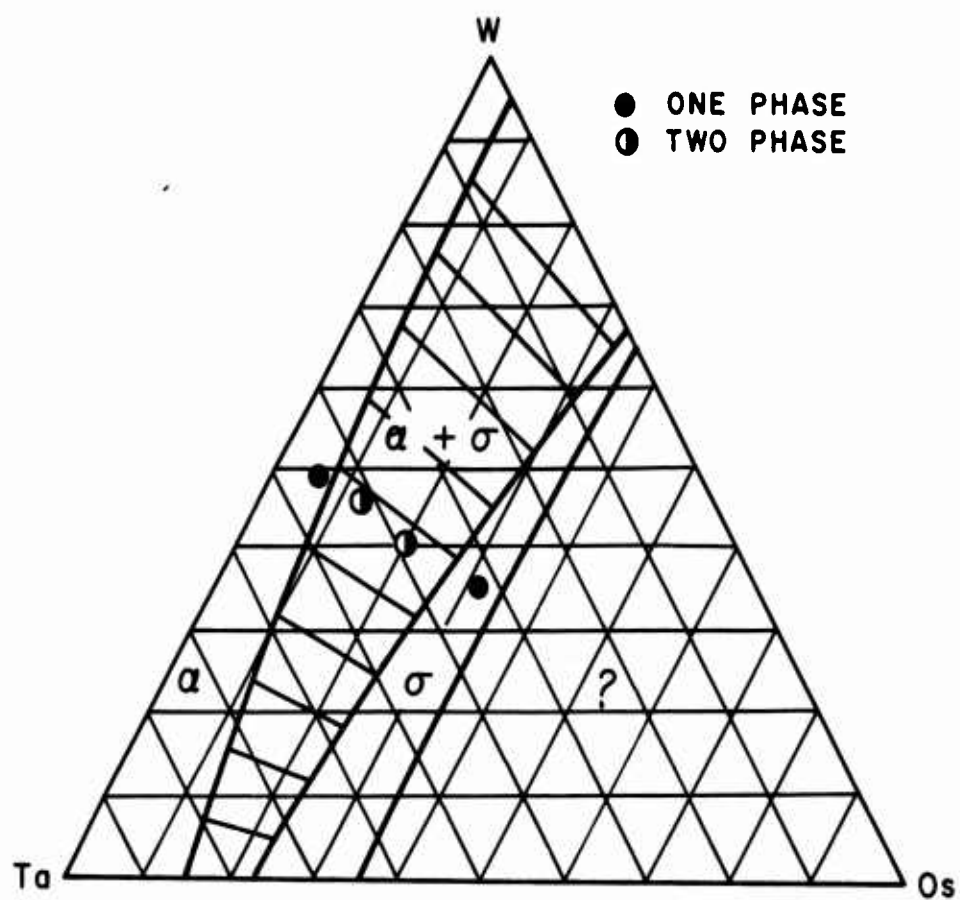
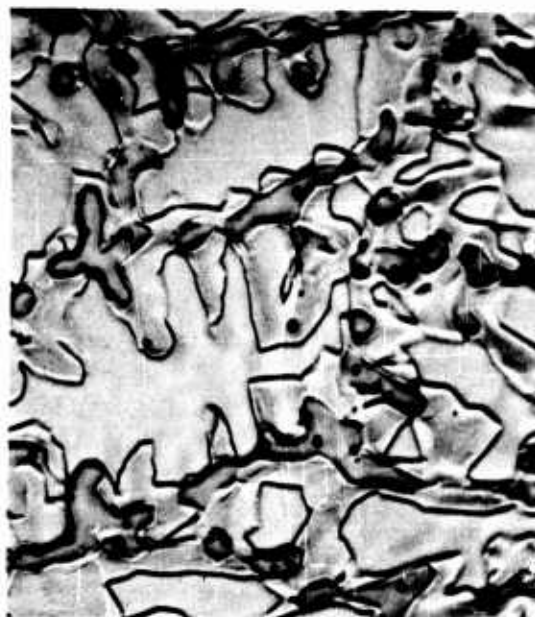


FIG. 24 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Ta-Os SYSTEM AT 1500° and 1000 °C.







Neg. No. 19109

X 1000

Fig. 26

As Cast Structure of an Alloy Containing  
25% W-15% Ta-60% Re Illustrating an  
Incomplete Peritectic Reaction. Etched  
with 60cc Glycerine, 20cc HF, 20cc HNO<sub>3</sub>,  
and 20cc H<sub>2</sub>O<sub>2</sub>.

#### 7. W-Mo-Nb System

Alloys containing:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 33% W | - | 34% Mo | - | 33% Nb |
| 57% W | - | 15% Mo | - | 28% Nb |
| 56% W | - | 29% Mo | - | 15% Nb |

confirmed the expectation of complete miscibility of the three components.

#### 8. W-Mo-V System

Alloys containing:

|         |   |        |   |         |
|---------|---|--------|---|---------|
| 18% W   | - | 77% Mo | - | 5% V    |
| 35% W   | - | 55% Mo | - | 10% V   |
| 38.5% W | - | 40% Mo | - | 21.5% V |
| 60% W   | - | 31% Mo | - | 9% V    |
| 76.5% W | - | 20% Mo | - | 3.5% V  |

all proved to be single-phase structures, leading to the conclusion that complete miscibility of the three components exists.

#### 9. W-Mo-Cr System

Two alloys containing:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 44% W | - | 20% Mo | - | 36% Cr |
| 51% W | - | 10% Mo | - | 39% Cr |

were made to check the location of the immiscibility zone emanating from the W-Cr binary system. This has been delineated by Grum and Grjimaýlo (41). Phase relationships at 1000°C are shown in Figure 27. At 1500°C complete solid solubility exists.

#### 10. W-Mo-Os System

Two ternary alloys:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 61% W | - | 32% Mo | - | 7% Os  |
| 57% W | - | 30% Mo | - | 13% Os |

combined with the binary solubility limits permit the location of the terminal solid solution limits shown in Figure 28. At very high temperatures the solubility limits must be somewhat higher than 30% Mo-13% Os because that alloy is single-phase in the as-cast condition.

#### 11. W-Mo-Re System

Three ternary alloys were melted:

|         |   |        |   |          |
|---------|---|--------|---|----------|
| 42% W   | - | 22% Mo | - | 36% Re   |
| 39.5% W | - | 15% Mo | - | 45.5% Re |
| 44% W   | - | 30% Mo | - | 26% Re   |

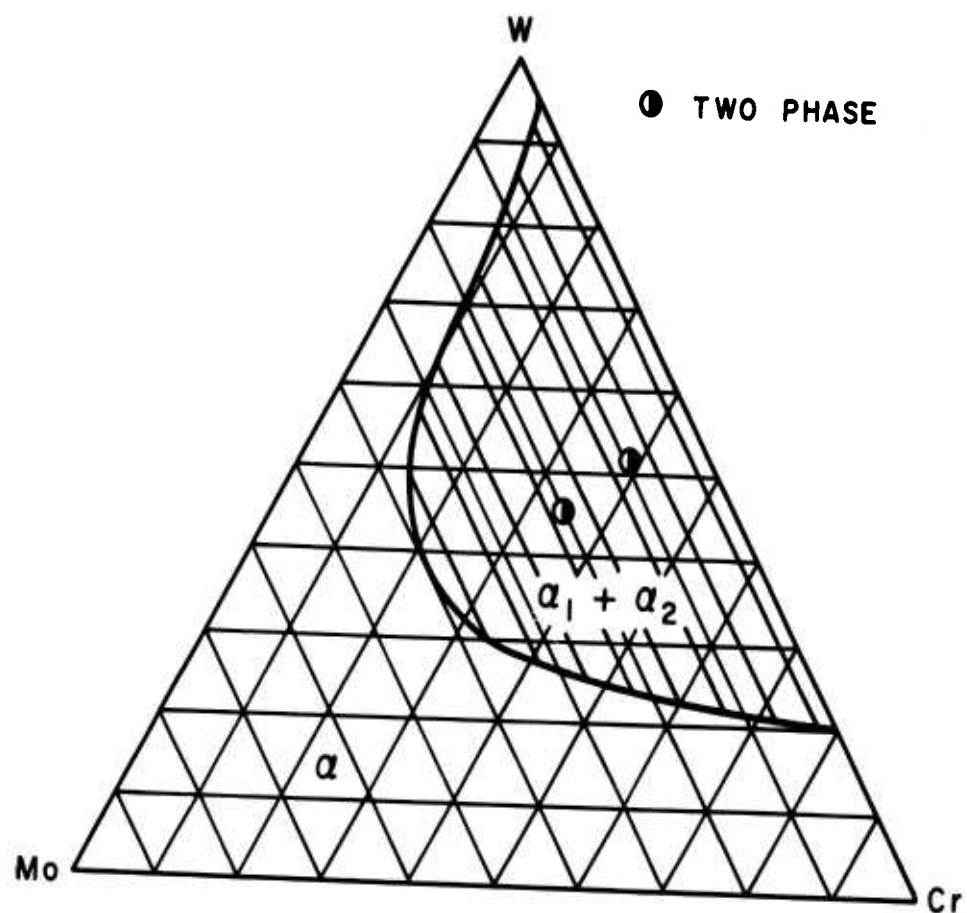


FIG. 27 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Mo-Cr SYSTEM AT 1000°C.

(Phase Boundary Shown Taken from  
Reference 41).

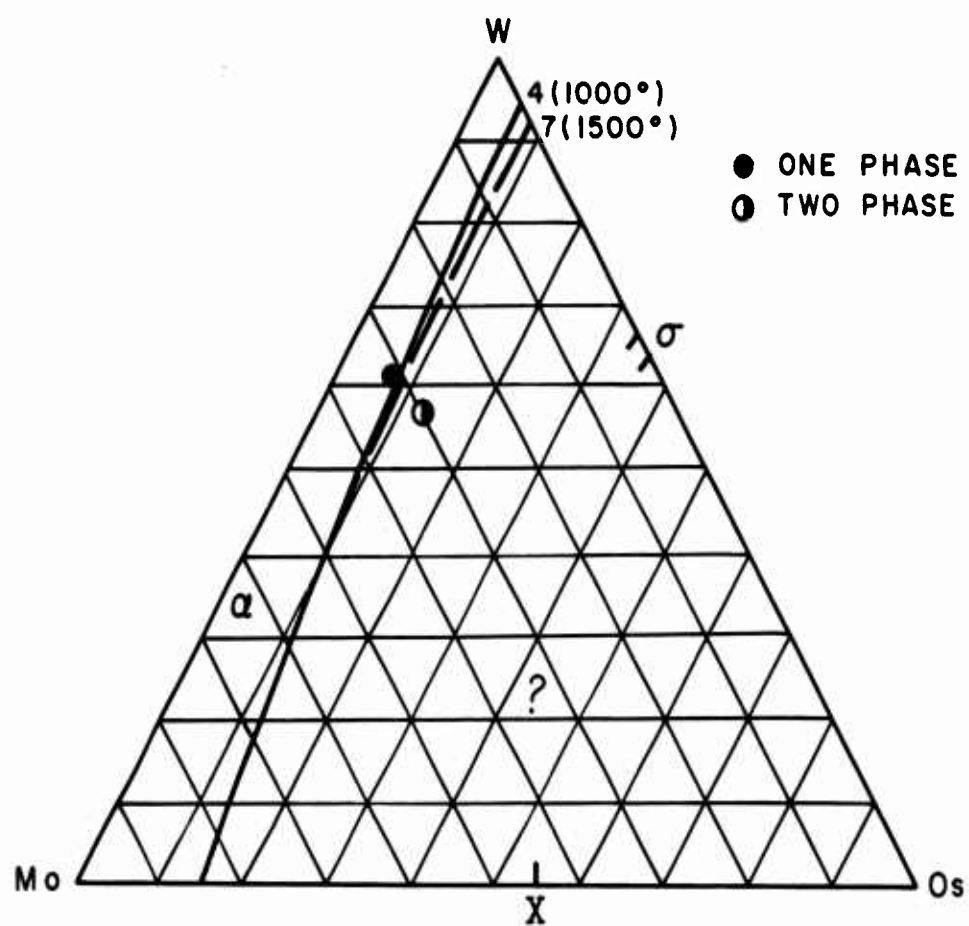


FIG. 28 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Mo-Os SYSTEM AT 1500° and 1000°C.

The solid solubility boundaries are shown in Figures 29 and 30. A considerable amount of twin-type banding was observed in the 30% Mo, 26% Re alloy.

#### 12. W-Nb-V System

Alloys were melted at the following compositions:

|       |   |          |   |         |
|-------|---|----------|---|---------|
| 39% W | - | 37% Nb   | - | 24% V   |
| 65% W | - | 16.5% Nb | - | 18.5% V |
| 61% W | - | 31% Nb   | - | 8% V    |

The single-phase structures observed confirmed the expectation that complete solid miscibility exists between three components.

#### 13. W-Nb-Cr System

The following alloys were arc melted:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 21% W | - | 61% Nb | - | 18% Cr |
| 40% W | - | 50% Nb | - | 10% Cr |
| 40% W | - | 40% Nb | - | 20% Cr |
| 48% W | - | 10% Nb | - | 42% Cr |

The as-cast structures of all four alloys showed the primary dendrite plus eutectic structure characteristic of the Nb-Cr system. The compound NbCr<sub>2</sub> is bonded strongly enough to push the solid solubility limits far back toward the tungsten corner as shown in Figure 31 and 32, phase relationships at 1500° and 1000°C, respectively.

#### 14. W-Nb-Os System

Seven alloys were melted of the following compositions:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 16% W | - | 67% Nb | - | 17% Os |
| 15% W | - | 54% Nb | - | 31% Os |
| 14% W | - | 43% Nb | - | 43% Os |
| 53% W | - | 33% Nb | - | 14% Os |
| 49% W | - | 25% Nb | - | 26% Os |
| 46% W | - | 18% Nb | - | 36% Os |
| 70% W | - | 18% Nb | - | 12% Os |

The resulting isothermal section in Figure 33 is valid both for 1500° and 1000°C. The 53% W-33% Nb-14% Os is just beyond the solid solubility limit as shown in Figure 34. The single-phase structure of the 46% W-18% Nb-36% Os alloy indicates that the first intermediate phase of the W-Os system penetrates deeply into the composition triangle.

#### 15. W-Nb-Re System

Two alloys containing:

|         |   |        |   |          |
|---------|---|--------|---|----------|
| 40% W   | - | 14% Nb | - | 46% Re   |
| 43.5% W | - | 25% Nb | - | 31.5% Re |

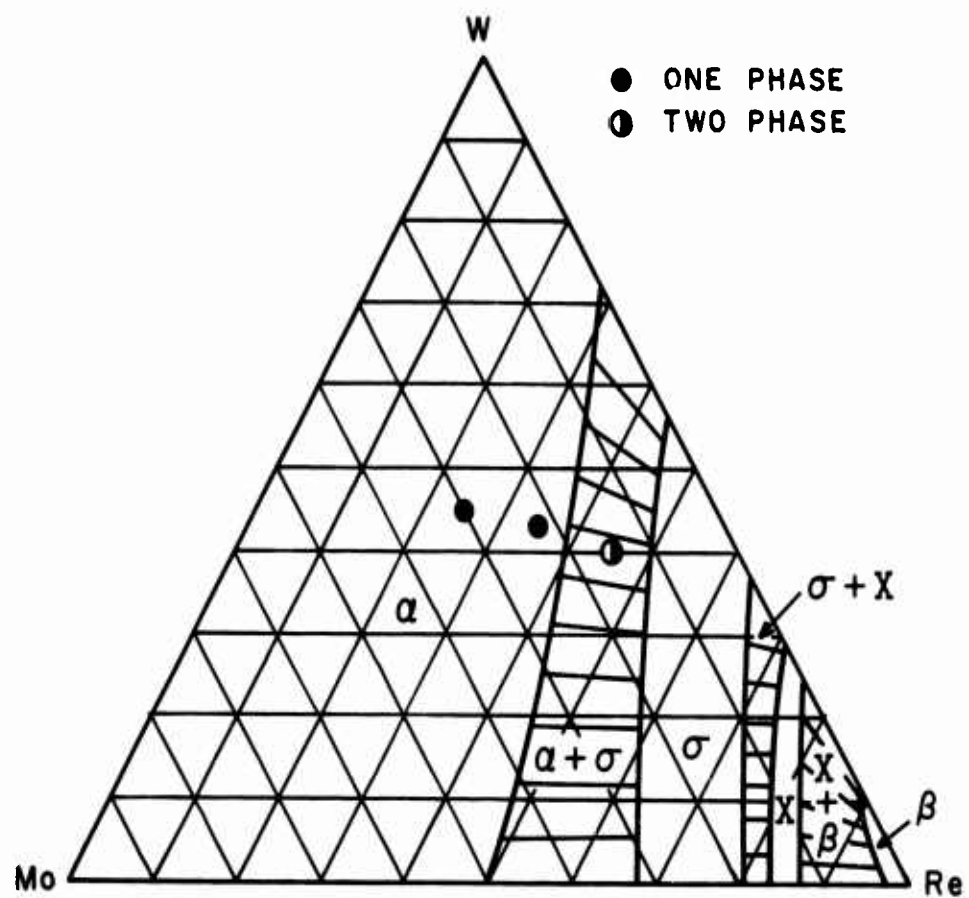


FIG. 29 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Mo-Re SYSTEM AT 1500°C.

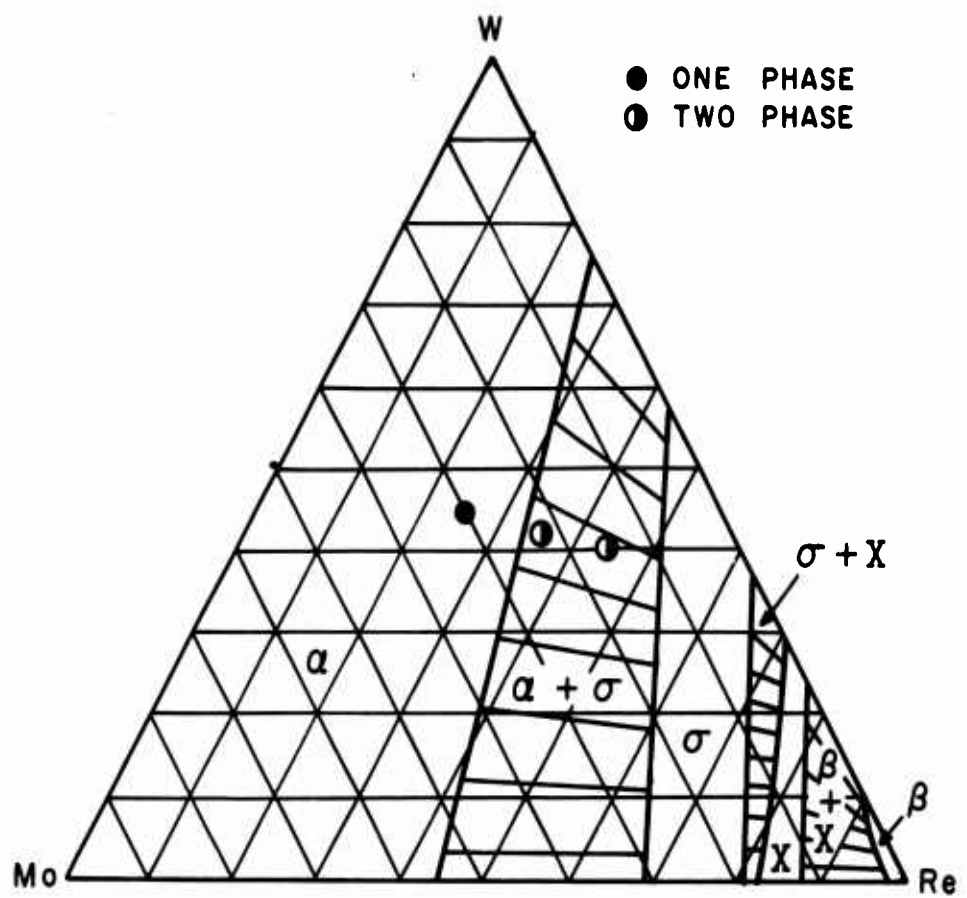


FIG. 30 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Mo-Re SYSTEM AT 1000°C.

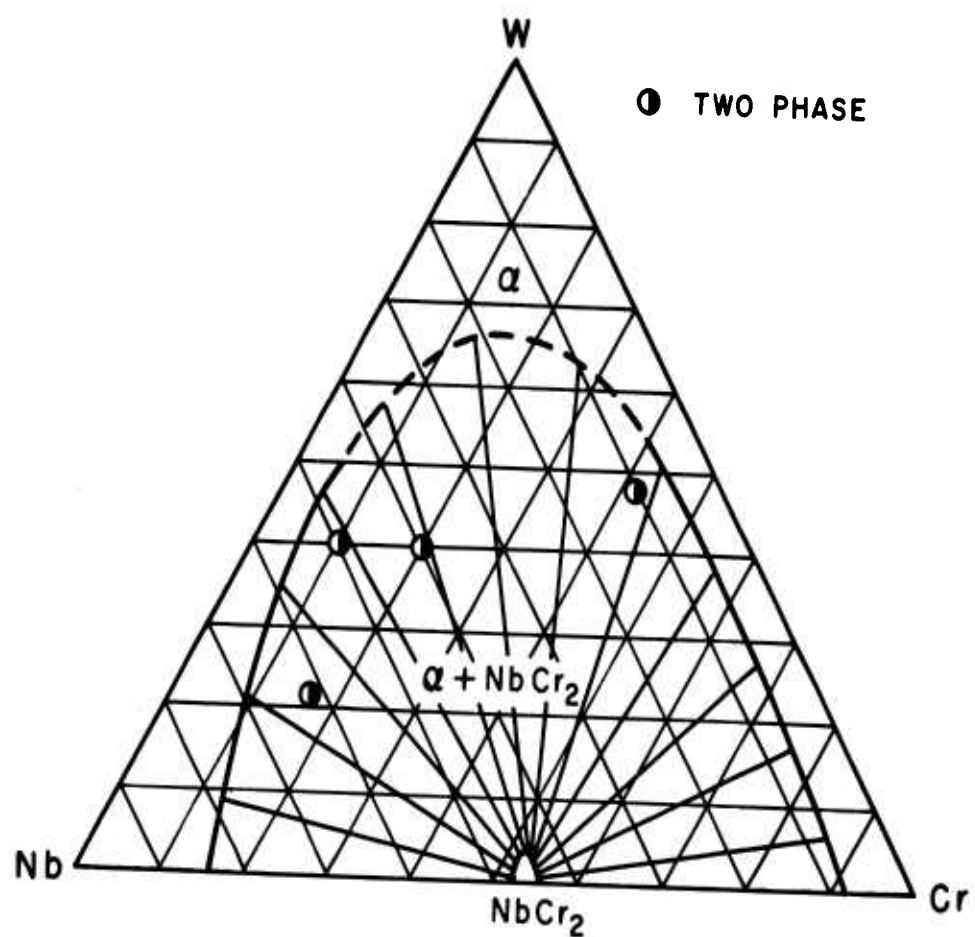


FIG. 31 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Nb-Cr SYSTEM AT 1500 °C.



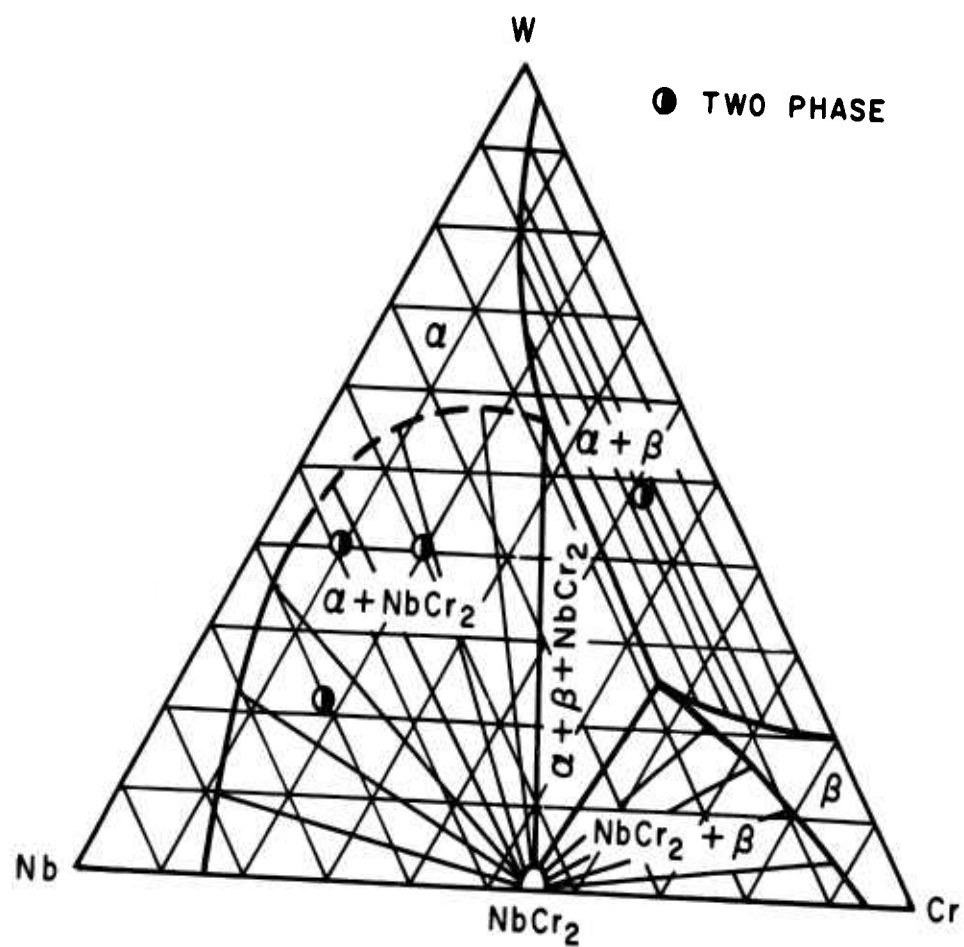


FIG. 32 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Nb-Cr SYSTEM AT 1000°C.

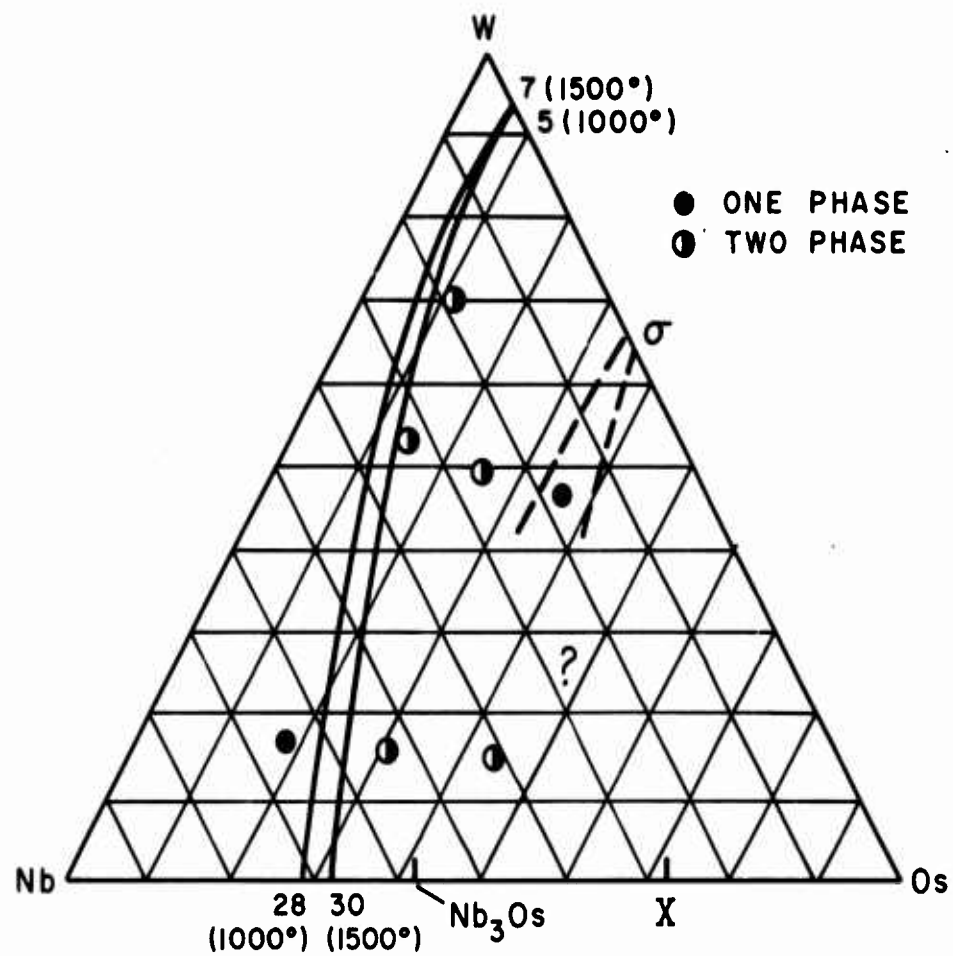
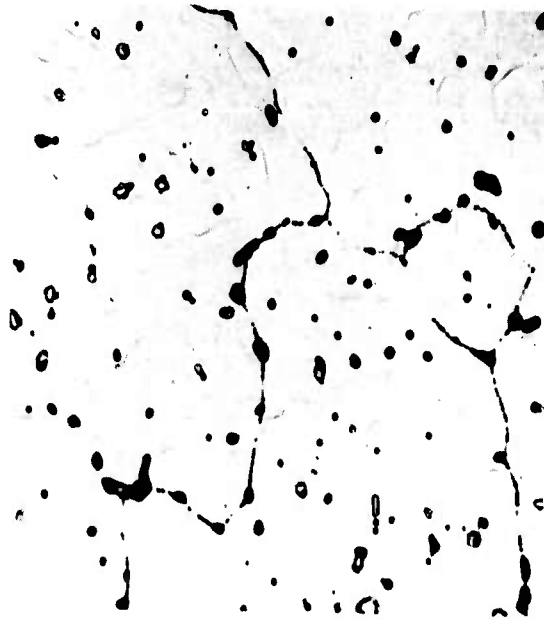


FIG. 33 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Nb-Os SYSTEM AT 1500° and 1000°C.



Neg. No. 18659

X 500

Fig. 34

Microstructure of a 53% W-33% Nb-14% Os Alloy Annealed at 1500°C for 3/4 hour Showing a Trace of Precipitation of a Second Phase. Electrolytically Etched with 5%  $H_2SO_4$ .

were sufficient to locate the boundary of the terminal solid solution shown in Figure 35.

#### 16. W-V-Cr System

Alloys containing:

|       |   |       |   |        |
|-------|---|-------|---|--------|
| 48% W | - | 10% V | - | 42% Cr |
| 46% W | - | 20% V | - | 34% Cr |

were prepared to locate the position of the boundary originating from immiscibility in the W-Cr system. Complete solid-state miscibility exists at 1500°C. The phase boundaries outlined at 1000°C with the aid of the two ternary alloys are shown in Figure 36.

#### 17. W-V-Os System

The three alloys melted contained:

|       |   |       |   |        |
|-------|---|-------|---|--------|
| 35% W | - | 29% V | - | 36% Os |
| 40% W | - | 40% V | - | 30% Os |
| 40% W | - | 40% V | - | 20% Os |

All three alloys fell in the terminal solid solution field indicating that the solubility boundary as shown in Figure 37 is probably a straight line joining the binary limits.

#### 18. W-V-Re System

Two alloys containing:

|       |   |       |   |        |
|-------|---|-------|---|--------|
| 30% W | - | 5% V  | - | 65% Re |
| 40% W | - | 10% V | - | 50% Re |

confirm the terminal solid solubility as approximately a straight line joining the binary limits as shown in Figure 38.

#### 19. W-Cr-Os System

Three alloys were melted containing:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 37% W | - | 38% Cr | - | 25% Os |
| 52% W | - | 38% Cr | - | 10% Os |
| 59% W | - | 36% Cr | - | 5% Os  |

After annealing at 1500°C all three compositions were observed to be single phase, thus confirming the extensive solid solubility on the W-Cr portion of the isotherm. The solubility of tungsten and osmium in chromium is 100 and 75%, respectively. After the 1000°C anneal, however, all alloys were two-phase and the tentative phase relationships at this temperature are presented in Figure 39.

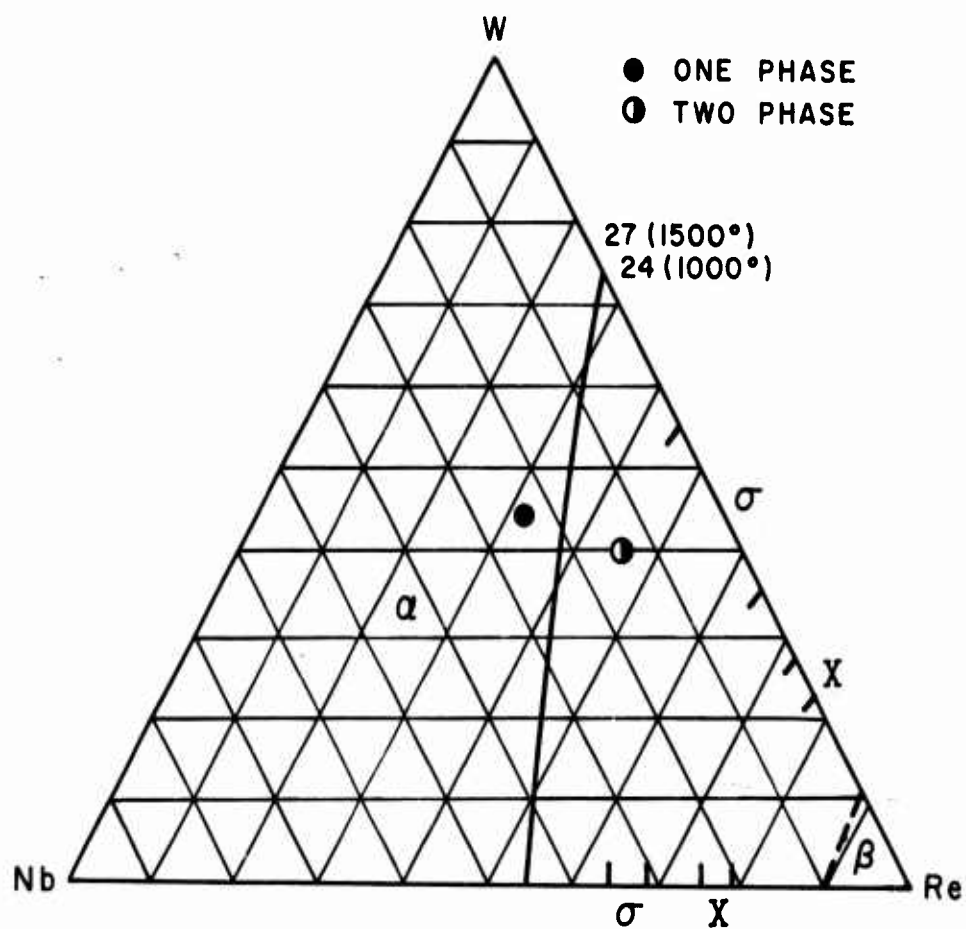


FIG. 35 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Nb-Re SYSTEM AT 1500° and 1000°C.

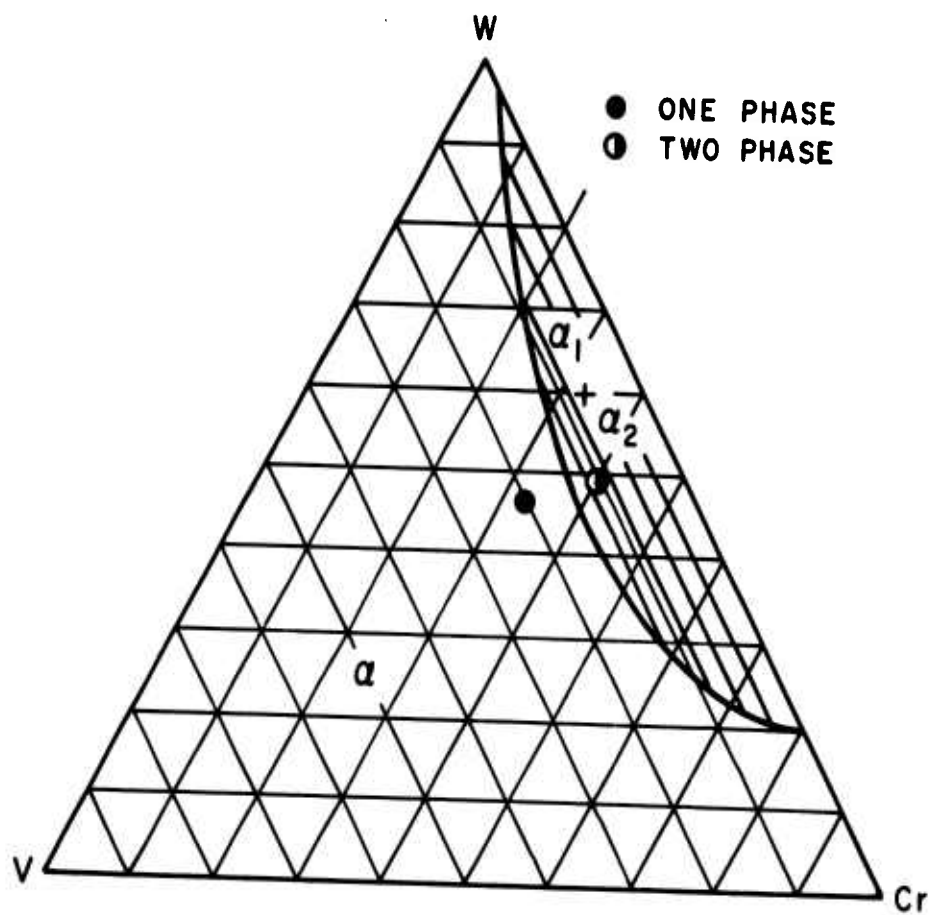


FIG. 36 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-V-Cr SYSTEM AT 1000°C.

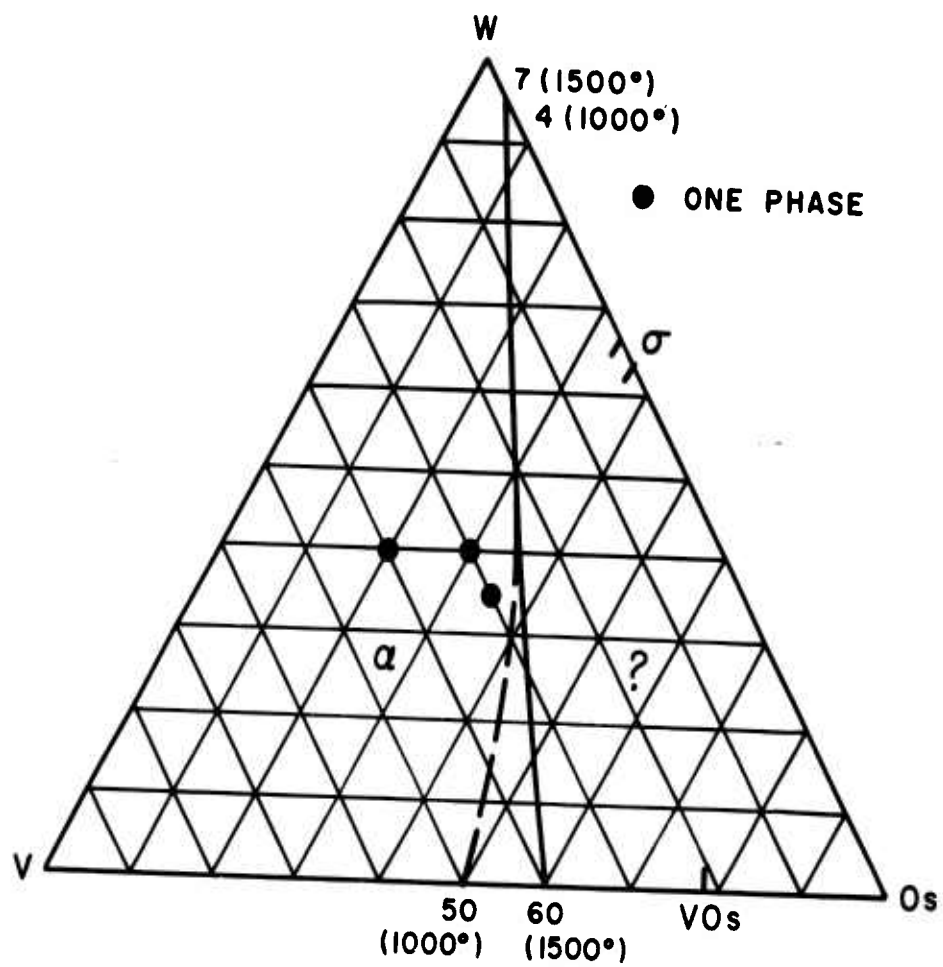


FIG. 37 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-V-Os SYSTEM AT 1500° and 1000°C.

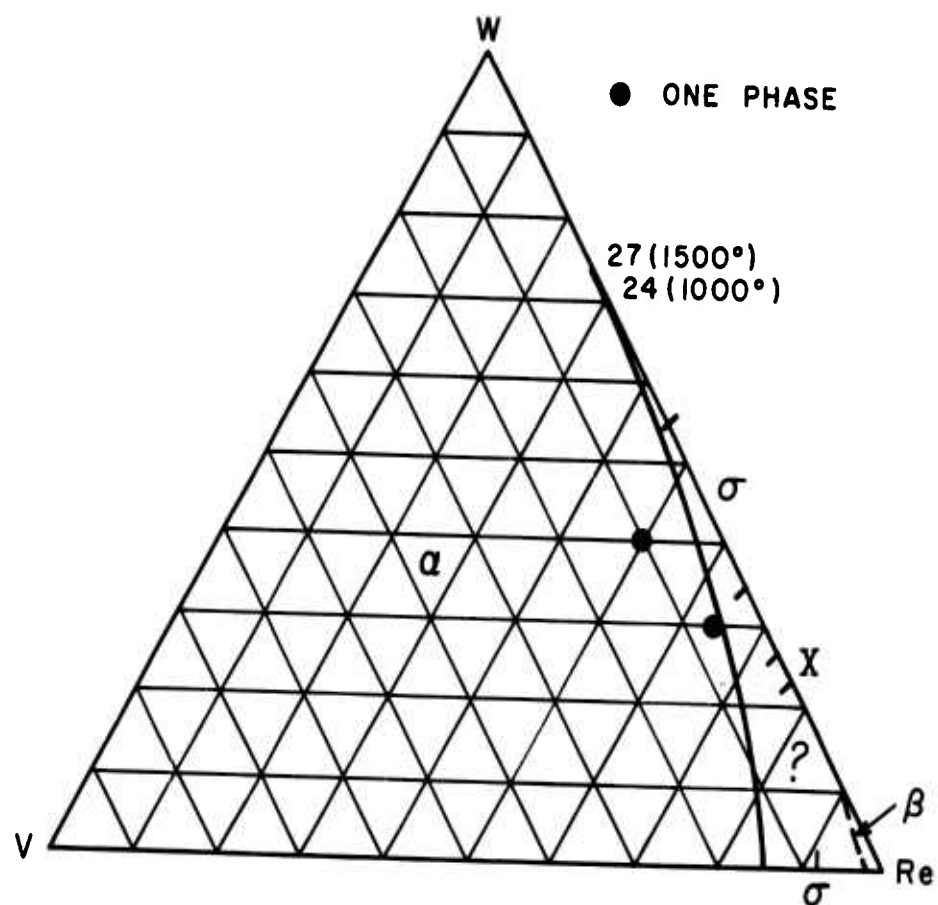


FIG. 38 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-V-Re SYSTEM AT 1500° and 1000°C.





## 20. W-Cr-Re System

Four alloys were arc melted to contain:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 18% W | - | 44% Cr | - | 38% Re |
| 40% W | - | 2% Cr  | - | 58% Re |
| 50% W | - | 30% Cr | - | 20% Re |
| 50% W | - | 40% Cr | - | 10% Re |

Phase relationships in this ternary system appear to be fairly complicated. A tentative isothermal section at 1500°C shown in Figure 40 serves to illustrate the broad W-Cr rich solid solution field. At 1000°C, however, the  $\alpha_1 + \alpha_2$  field of the W-Cr system extends into this solid solution (Figure 41).

## 21. W-Os-Re System

Two alloys containing:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 90% W | - | 5% Os  | - | 5% Re  |
| 80% W | - | 10% Os | - | 10% Re |

demonstrate the limited zone of the tungsten-base solid solutions. The section is shown in Figure 42. Osmium and rhenium form a continuous series of solid solutions, but this field has not been fully shown in the figure because the solubility of tungsten in osmium is unknown.

## 22. Ta-Mo-Nb System

Three alloys were melted:

|       |   |        |   |        |
|-------|---|--------|---|--------|
| 32% W | - | 34% Mo | - | 34% Nb |
| 56% W | - | 15% Mo | - | 29% Nb |
| 56% W | - | 30% Mo | - | 14% Nb |

These all had single-phase structures supporting the expectation that complete miscibility exists between the three components.

## 23. Ta-Mo-V System

Five alloys containing the following compositions were melted:

|        |   |        |   |       |
|--------|---|--------|---|-------|
| 35% Ta | - | 40% Mo | - | 25% V |
| 40% Ta | - | 15% Mo | - | 45% V |
| 45% Ta | - | 10% Mo | - | 45% V |
| 65% Ta | - | 17% Mo | - | 18% V |
| 60% Ta | - | 32% Mo | - | 8% V  |

At 1500°C all alloys were single-phase as anticipated. Phase relationships at 1000°C are depicted in Figure 43. The 45% Ta-10% Mo alloy annealed at 1000°C had about 5% second phase and was therefore adjudged to lie right on the phase boundary as shown.

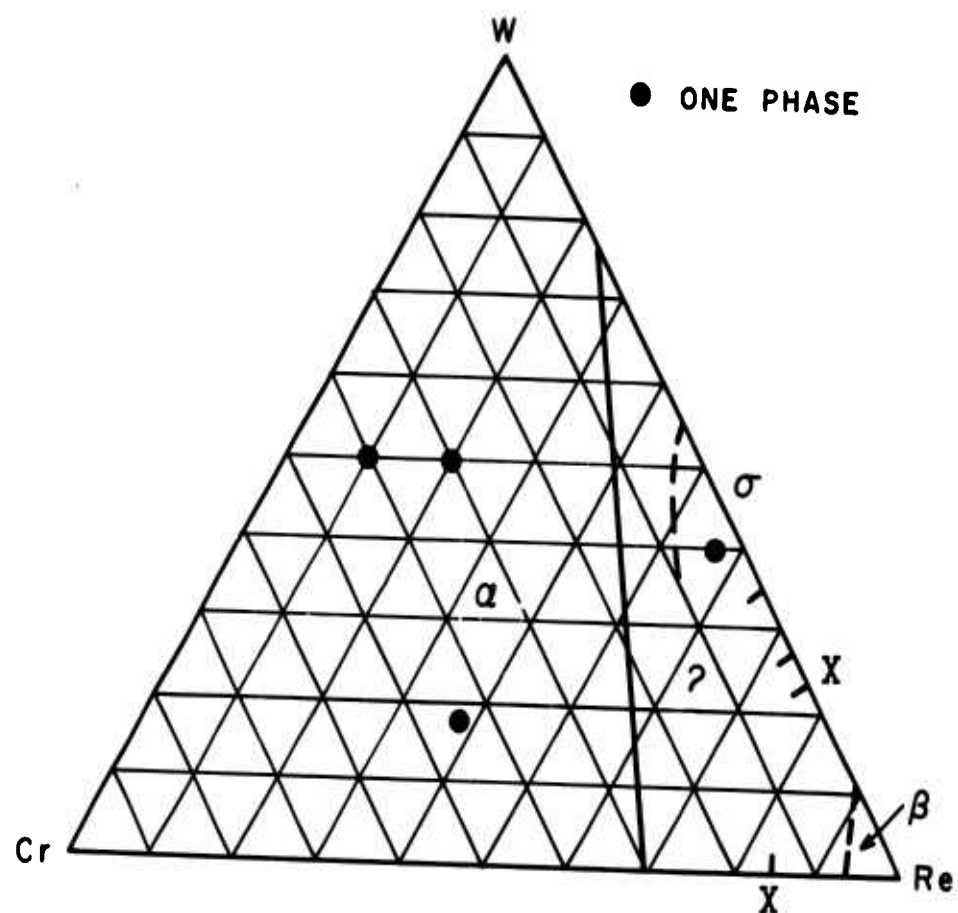


FIG. 40 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Cr-Re SYSTEM AT 1500°C.



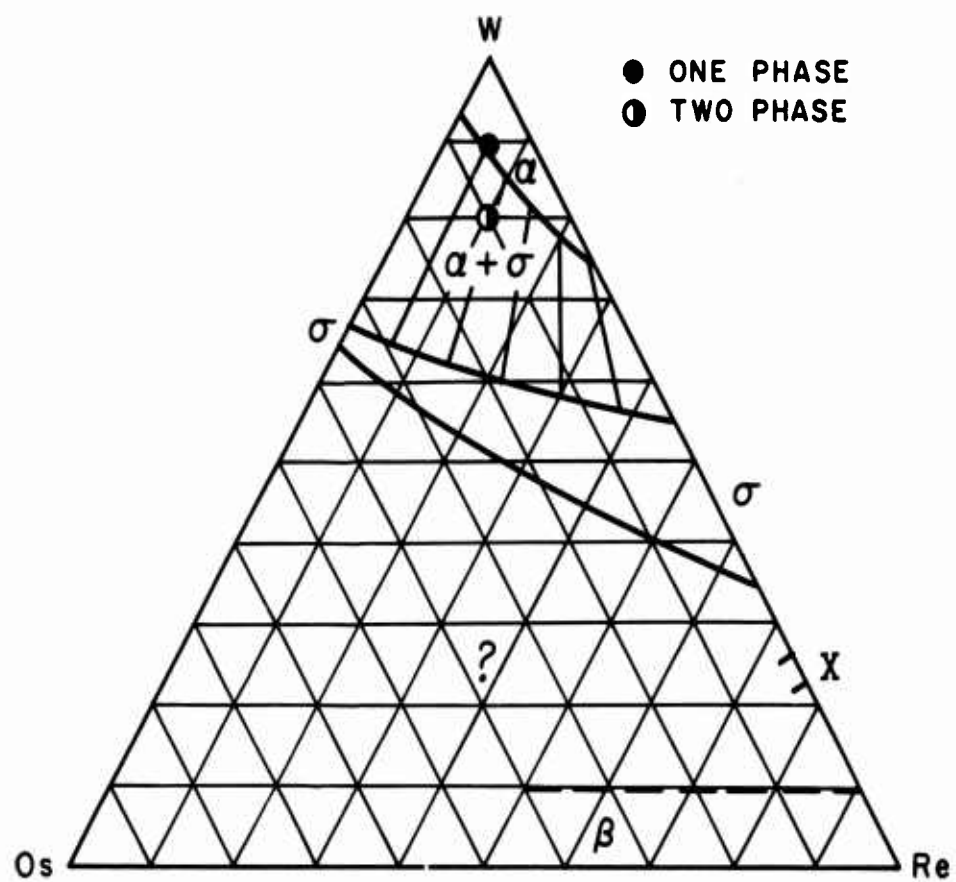


FIG. 42 - TENTATIVE PHASE RELATIONSHIPS  
OF THE W-Os-Re SYSTEM AT 1500° and 1000°C.

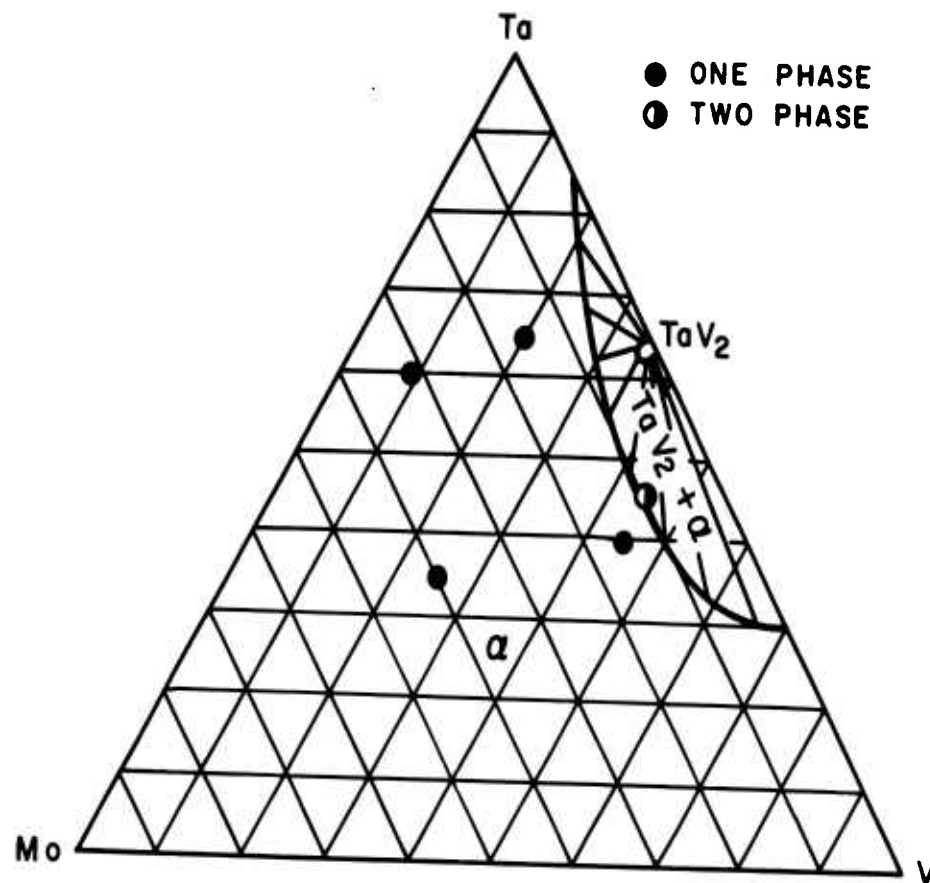


FIG. 43 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Mo-V SYSTEM AT 1000 °C.

#### 24. Ta-Mo-Cr System

Four alloys were arc melted containing:

|        |   |        |   |        |
|--------|---|--------|---|--------|
| 42% Ta | - | 23% Mo | - | 35% Cr |
| 40% Ta | - | 20% Mo | - | 40% Cr |
| 40% Ta | - | 30% Mo | - | 30% Cr |
| 40% Ta | - | 10% Mo | - | 50% Cr |

All alloys showed as-cast structures containing primary dendrites plus eutectic characteristic of the Ta-Cr system. In the annealed condition all alloys were found to be two-phase. Based on the information from the four compositions, the sections at 1500° and 1000°C are shown in Figure 44.

#### 25. Ta-Mo-Os System

Three alloys were melted containing:

|        |   |        |   |        |
|--------|---|--------|---|--------|
| 37% Ta | - | 26% Mo | - | 37% Os |
| 49% Ta | - | 26% Mo | - | 25% Os |
| 37% Ta | - | 47% Mo | - | 16% Os |

These indicate that the total solid solubility in the ternary system exceeds either binary. The common isothermal section is shown in Figure 45.

#### 26. Ta-Mo-Re System

Three alloys containing:

|          |   |        |   |          |
|----------|---|--------|---|----------|
| 34.5% Ta | - | 18% Mo | - | 47.5% Re |
| 23% Ta   | - | 18% Mo | - | 59% Re   |
| 20% Ta   | - | 30% Mo | - | 50% Re   |

combined with the data on the binary systems define the two-phase region ( $\alpha + \sigma$ ) in Figure 46. The 23% Ta-18% Mo-59% Re and the 20% Ta-30% Mo-50% Re alloys are nearly at the  $\alpha + \sigma/\sigma$  boundary according to the proportions of phases observed.

#### 27. Ta-Nb-V System

Five alloys were prepared containing:

|        |   |          |   |         |
|--------|---|----------|---|---------|
| 39% Ta | - | 40% Nb   | - | 21% V   |
| 65% Ta | - | 17% Nb   | - | 18% V   |
| 56% Ta | - | 14.5% Nb | - | 29.5% V |
| 25% Ta | - | 26% Nb   | - | 49% V   |
| 36% Ta | - | 12% Nb   | - | 52% V   |

At 1500°C, all alloys were single-phase indicating, as expected, complete miscibility of the three components. At 1000°C, the two phase fields generated by the formation of TaV<sub>2</sub> extend into the ternary section as shown in Figure 47. The 65% Ta-17% Nb-18% V alloy is almost on the phase boundary.

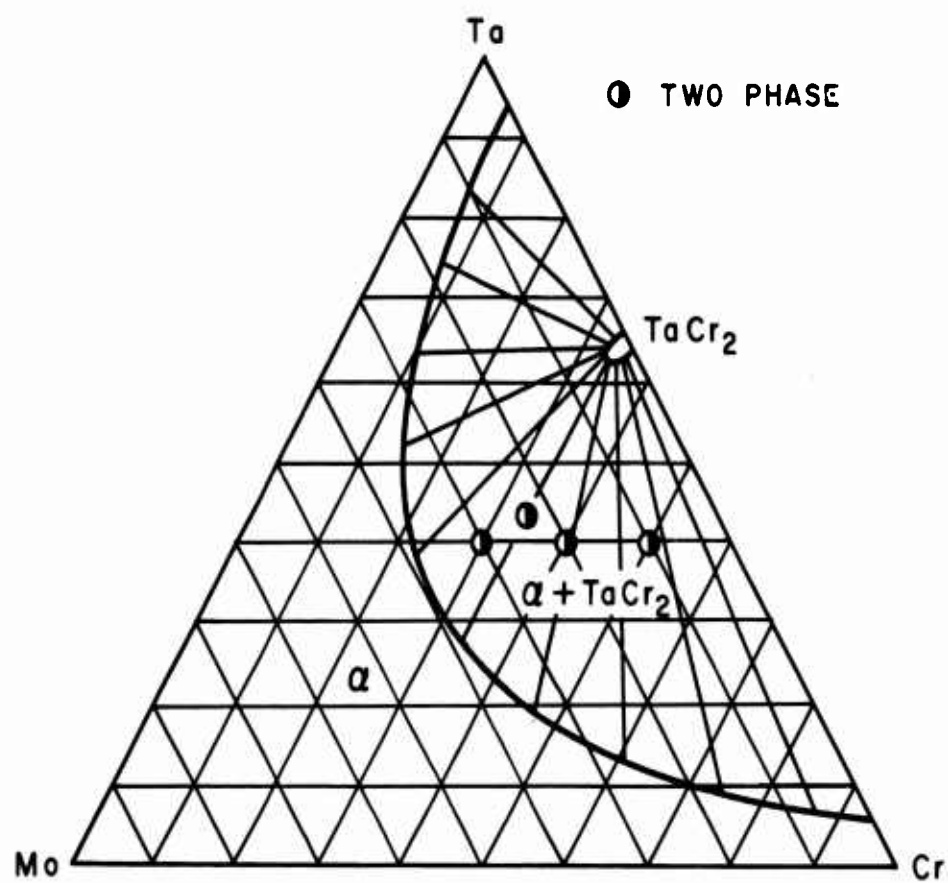


FIG. 44 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Mo-Cr SYSTEM AT 1500° and 1000°C.



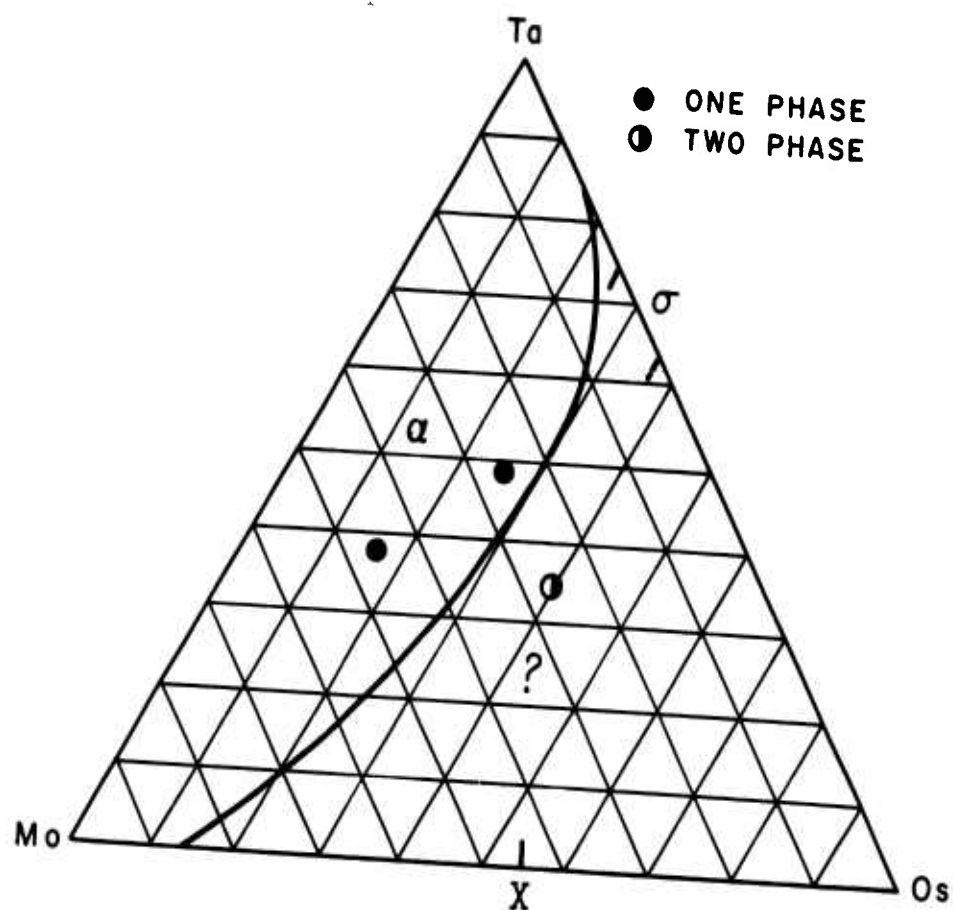


FIG. 45 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Mo-Os SYSTEM AT 1500° and 1000°C.

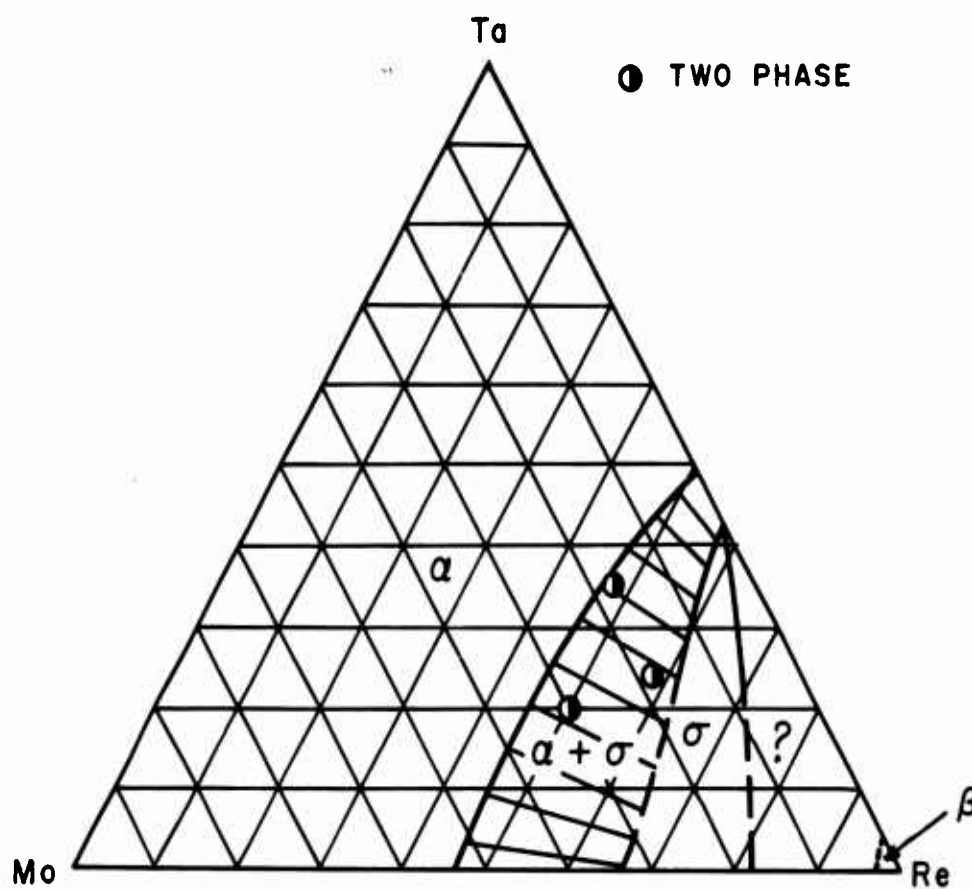


FIG. 46 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Mo-Re SYSTEM AT 1500° and 1000°C.\*

\*See footnote, Page 15

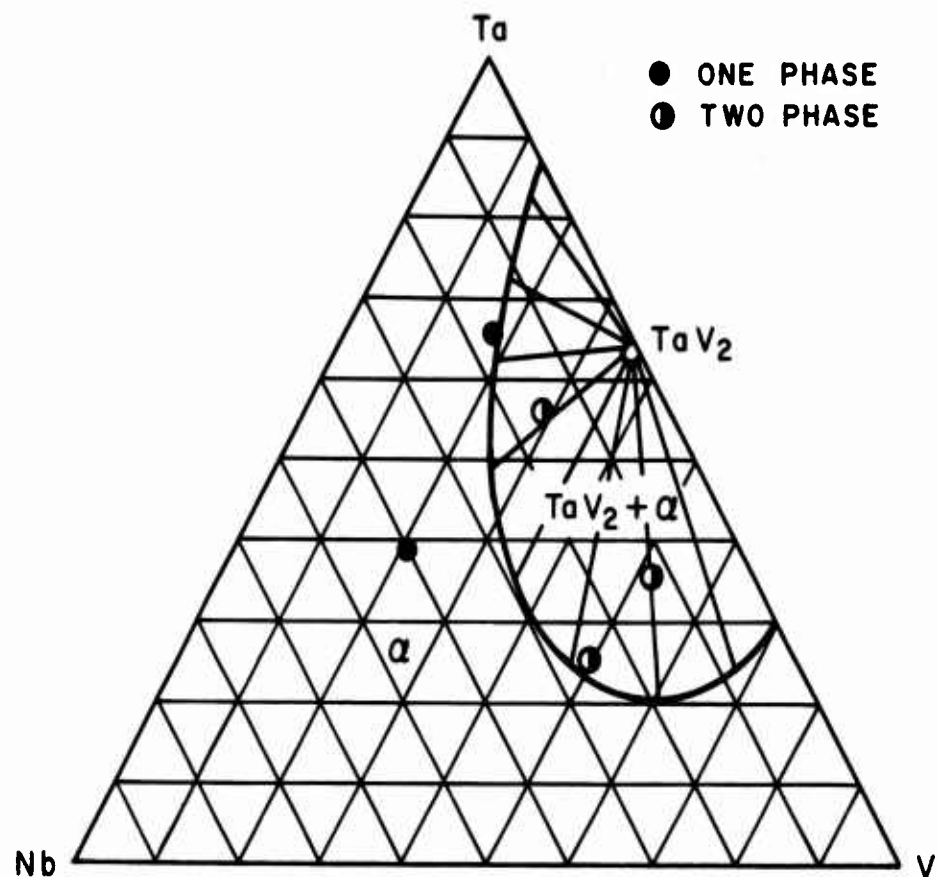


FIG. 47 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Nb-V SYSTEM AT 1000 °C.

## 28. Ta-Nb-Cr System

Six alloys were prepared containing:

|        |   |        |   |        |
|--------|---|--------|---|--------|
| 52% Ta | - | 44% Nb | - | 4% Cr  |
| 83% Ta | - | 14% Nb | - | 3% Cr  |
| 32% Ta | - | 32% Nb | - | 36% Cr |
| 18% Ta | - | 56% Nb | - | 26% Cr |
| 47% Ta | - | 38% Nb | - | 15% Cr |
| 40% Ta | - | 40% Nb | - | 20% Cr |

The first two alloys solidified as single phase and remained so on annealing at 1500°C. After the 1000°C anneal, the 52% Ta-44% Nb-4% Cr alloy showed a very small amount of precipitation from solid solution. The last three alloys showed primary dendrites plus eutectic characteristic of both the Ta-Cr and Nb-Cr systems in the cast structures. They did not change significantly upon annealing at 1500° or 1000°C.

Since NbCr<sub>2</sub> and TaCr<sub>2</sub> are structurally related Laves phases, it may be expected that they will be miscible to a large extent, if not completely, across the ternary system. These points are illustrated in Figure 48.

## 29. Ta-Nb-Os System

Of the four alloys arc melted:

|          |   |          |   |          |
|----------|---|----------|---|----------|
| 39% Ta   | - | 33.5% Nb | - | 27.5% Os |
| 66% Ta   | - | 11% Nb   | - | 23% Os   |
| 52.5% Ta | - | 34% Nb   | - | 13.5% Os |
| 77% Ta   | - | 11% Nb   | - | 12% Os   |

the first two closely defined the terminal solid solubility limit. The 66% Ta-11% Nb-23% Os alloy is just over the boundary according to the small proportion of second phase in the microstructures. Phase relationships at the two temperatures studied are shown in Figure 49.

## 30. Ta-Nb-Re System

Two alloys at:

|        |   |        |   |        |
|--------|---|--------|---|--------|
| 22% Ta | - | 14% Nb | - | 64% Re |
| 35% Ta | - | 18% Nb | - | 47% Re |

indicate, as shown in Figure 50, that the solid solubility limit is roughly a straight line joining the binary limits.

## 31. Ta-V-Cr System

Four alloys used to describe this system:

|        |   |       |   |        |
|--------|---|-------|---|--------|
| 89% Ta | - | 8% V  | - | 3% Cr  |
| 84% Ta | - | 11% V | - | 5% Cr  |
| 26% Ta | - | 35% V | - | 39% Cr |
| 15% Ta | - | 46% V | - | 39% Cr |

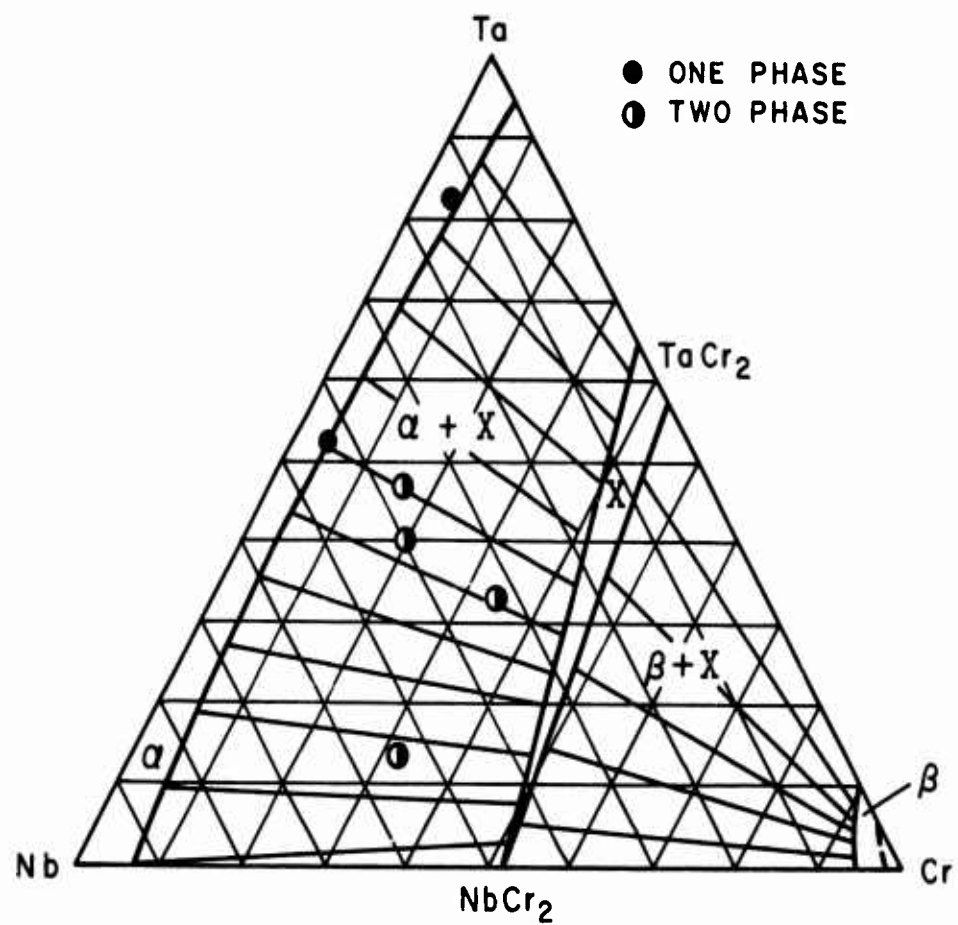


FIG. 48 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Nb-Cr SYSTEM AT 1500° and 1000°C.

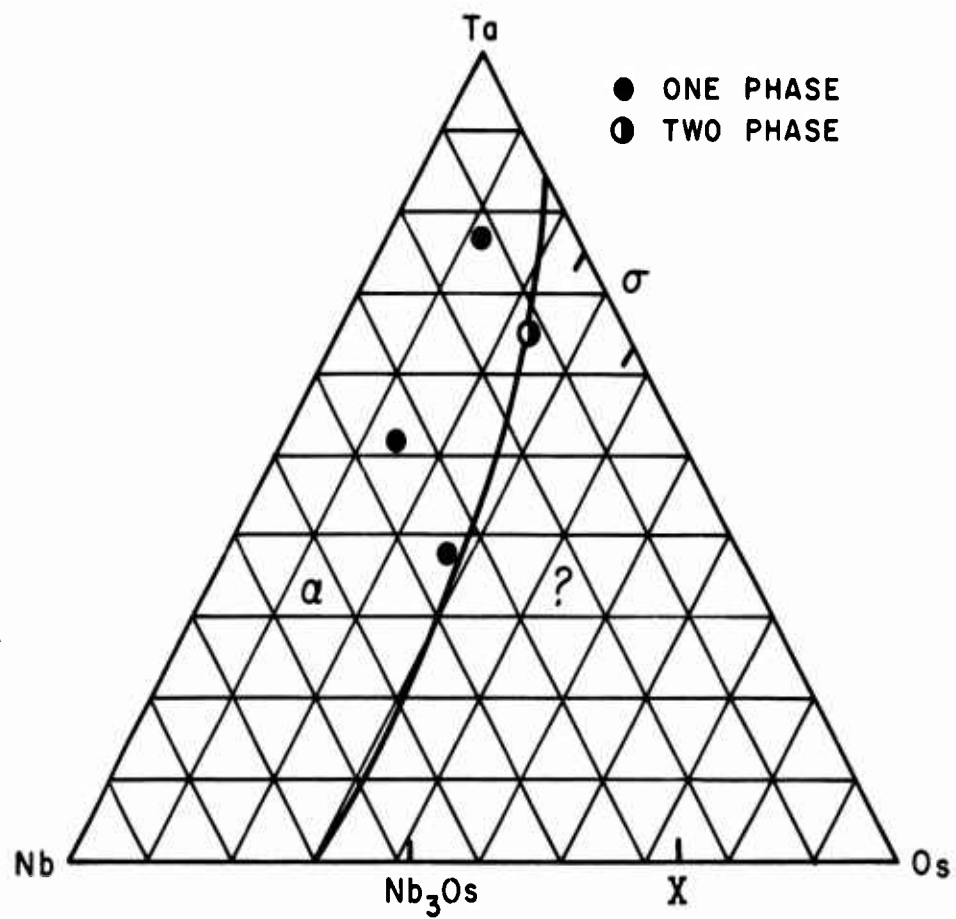


FIG. 49 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Nb-Os SYSTEM AT 1500° and 1000°C.

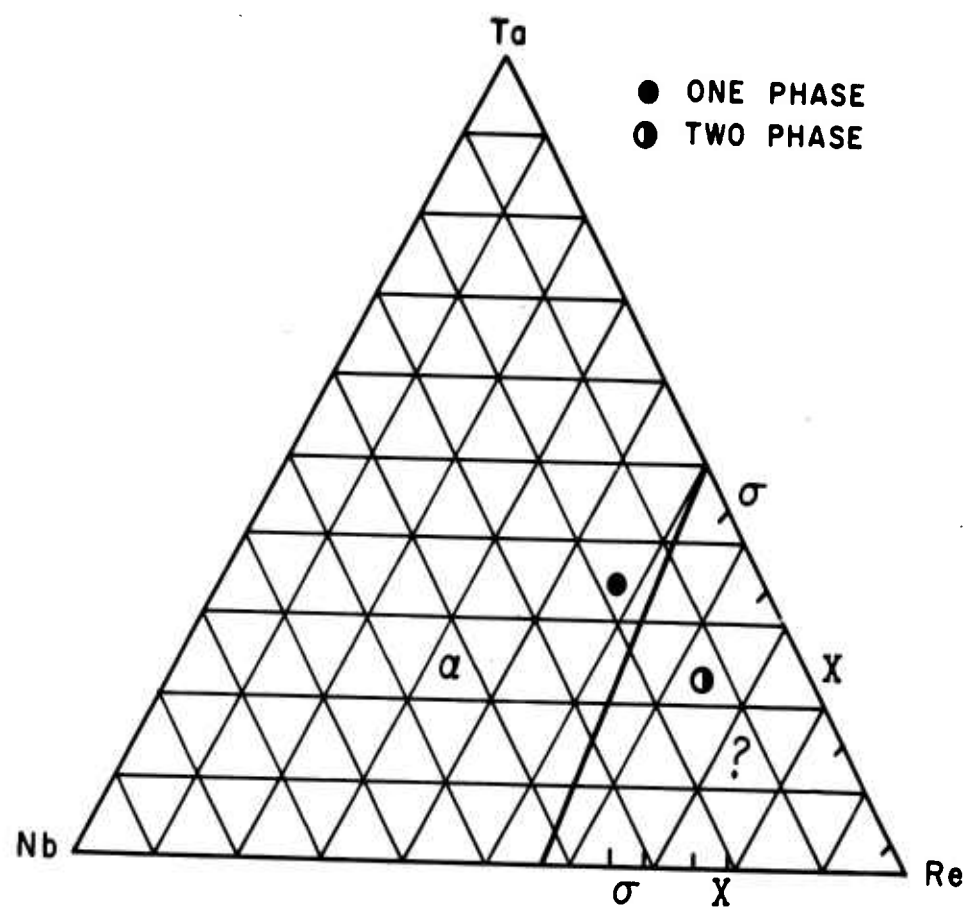


FIG. 50 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Nb-Re SYSTEM AT 1500° and 1000°C.\*

\*See footnote, Page 15

As with other systems,  $\text{TaCr}_2$  forces the large two-phase field ( $\alpha + \text{TaCr}_2$ ) to form at  $1500^\circ\text{C}$ , shown in Figure 51. It appears that at  $1000^\circ\text{C}$  (Figure 52), a two-phase field is generated between the two Laves-type phases  $\text{TaV}_2$  and  $\text{TaCr}_2$ , causing the existence of a three-phase field ( $\text{Ta} + \text{TaV}_2 + \text{TaCr}_2$ ).

### 32. Ta-V-Os System

Five alloys were used to define the system:

|        |   |        |   |          |
|--------|---|--------|---|----------|
| 86% Ta | - | 3% V   | - | 11% Os   |
| 81% Ta | - | 6.5% V | - | 12.5% Os |
| 75% Ta | - | 3% V   | - | 22% Os   |
| 50% Ta | - | 40% V  | - | 10% Os   |
| 30% Ta | - | 40% V  | - | 30% Os   |

At  $1500^\circ\text{C}$  all the alloys were single-phase, indicating that the solid solution field is very nearly defined by a straight line connecting the solubilities in the binary systems. This is shown in Figure 53. Phase relationships at  $1000^\circ\text{C}$  are slightly more complicated, as depicted in Figure 54.

### 33. Ta-V-Re System

Six alloys were prepared to study this system:

|        |   |       |   |        |
|--------|---|-------|---|--------|
| 86% Ta | - | 3% V  | - | 11% Re |
| 64% Ta | - | 3% V  | - | 33% Re |
| 76% Ta | - | 11% V | - | 13% Re |
| 50% Ta | - | 11% V | - | 39% Re |
| 45% Ta | - | 35% V | - | 20% Re |
| 20% Ta | - | 10% V | - | 70% Re |

After annealing at  $1500^\circ\text{C}$ , all but one of the compositions was single-phase. Figure 55 depicts the solid solution field at this temperature. At  $1000^\circ\text{C}$  the occurrence of  $\text{TaV}_2$  introduces the two-phase field, and this is shown in Figure 56.

### 34. Ta-Cr-Os System

Two alloys were prepared to define the solubility limits in this system:

|          |   |         |   |         |
|----------|---|---------|---|---------|
| 95.5% Ta | - | 2% Cr   | - | 2.5% Os |
| 85% Ta   | - | 7.5% Cr | - | 7.5% Os |

These two compositions served to bracket the tantalum-rich solid state solubility at  $1500^\circ$  and  $1000^\circ\text{C}$  adequately. Tentative phase relationships are shown in Figure 57.

### 35. Ta-Cr-Re System

Two ternary compositions were arc melted:

|        |   |        |   |        |
|--------|---|--------|---|--------|
| 72% Ta | - | 10% Cr | - | 18% Re |
| 61% Ta | - | 4% Cr  | - | 35% Re |



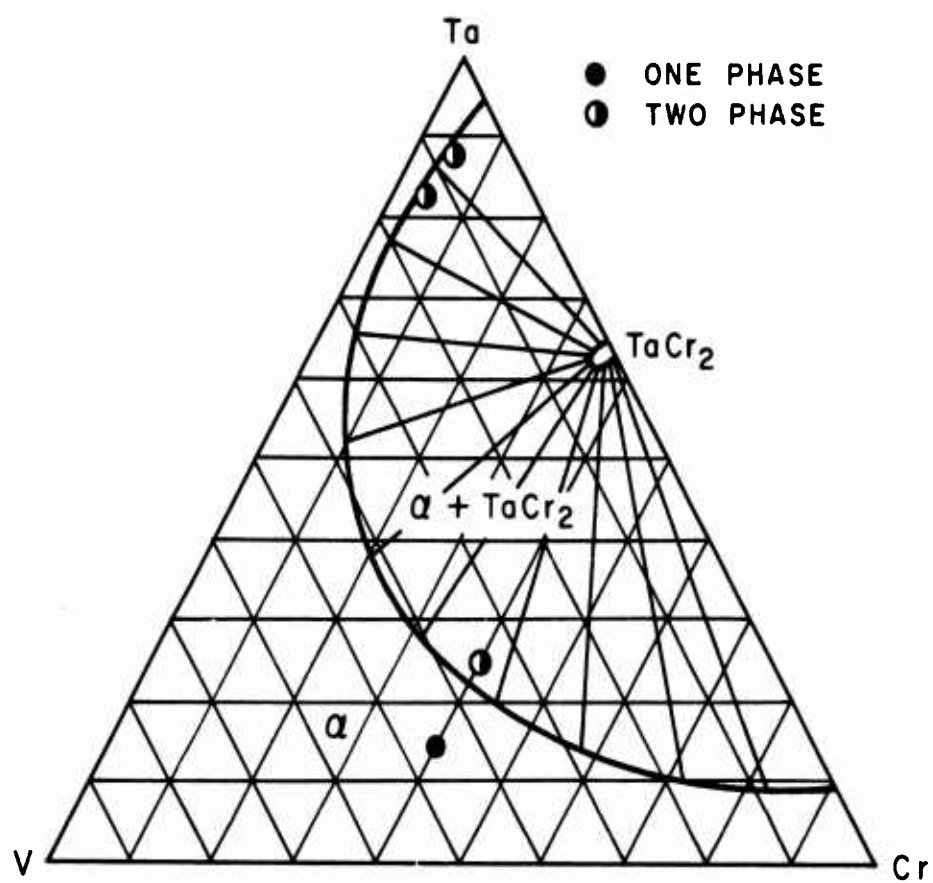


FIG. 51 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-V-Cr SYSTEM AT 1500°C.

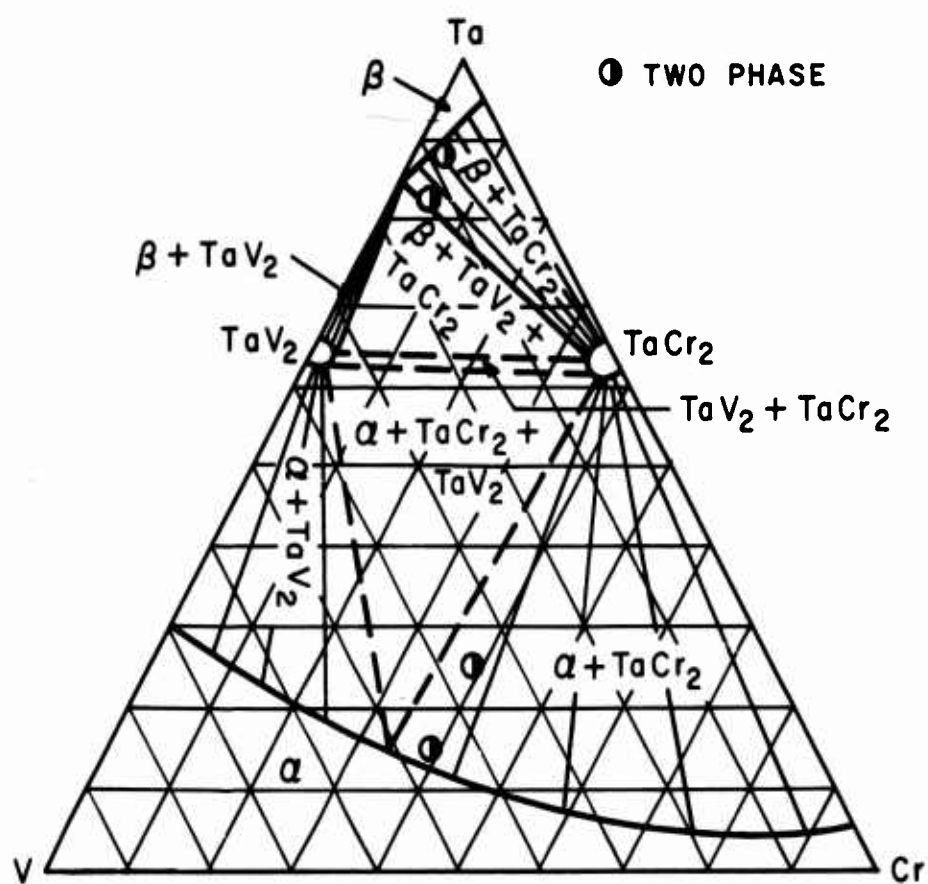


FIG. 52 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-V-Cr SYSTEM AT 1000 °C.

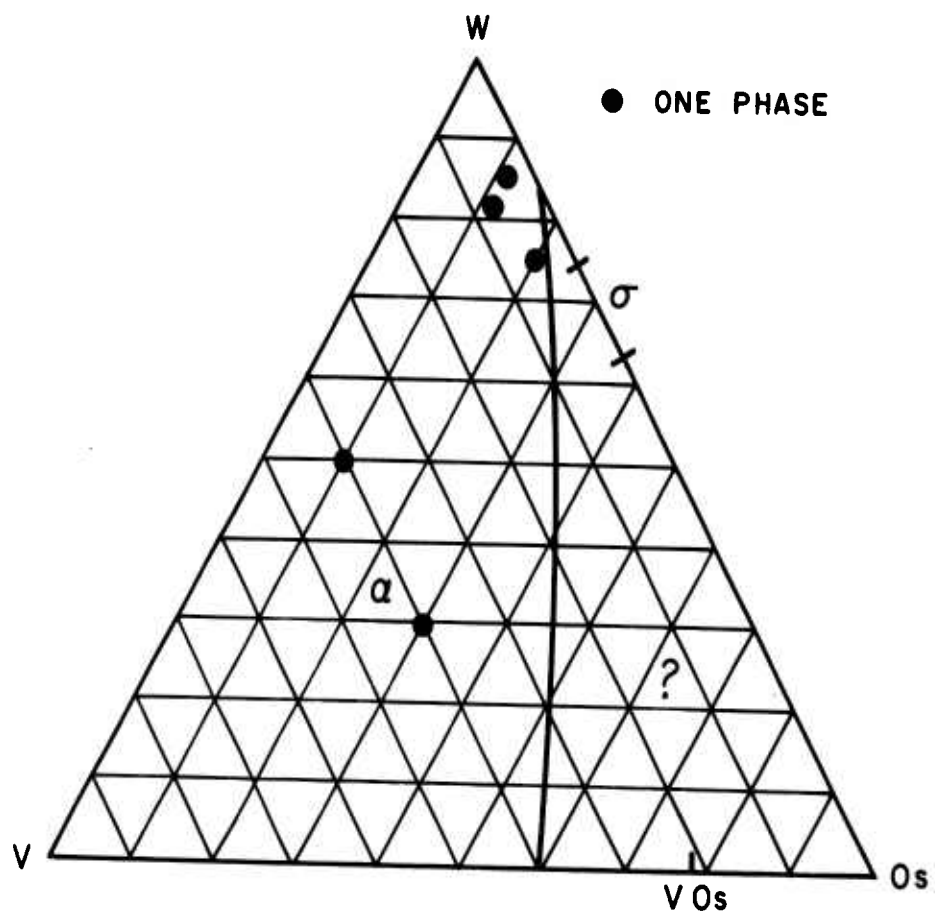


FIG. 53 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-V-Os SYSTEM AT 1500 °C.

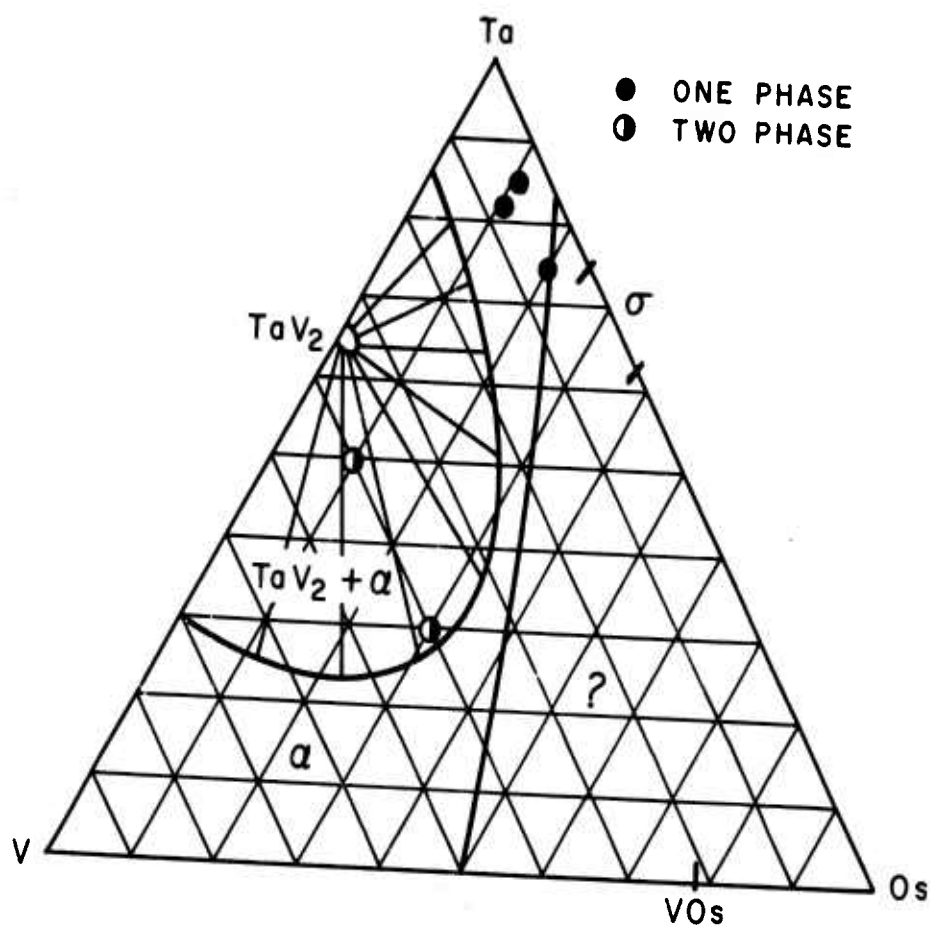


FIG. 54 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-V-Os SYSTEM AT 1000°C.

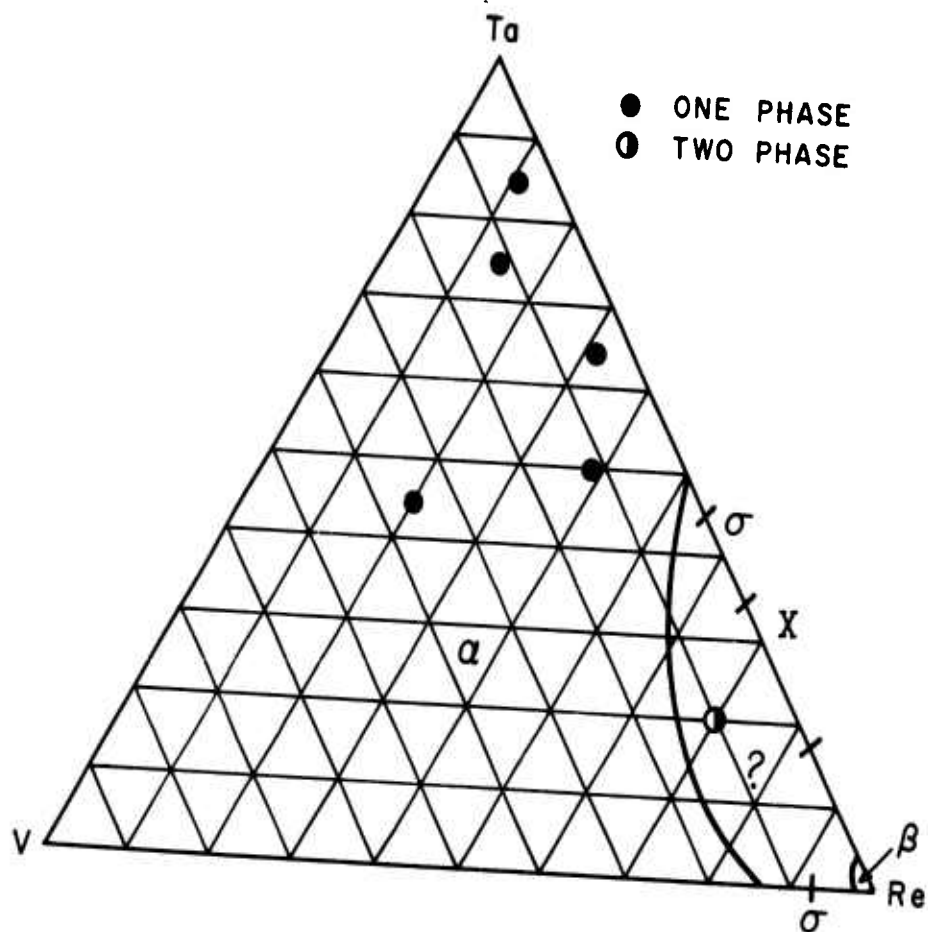


FIG. 55 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-V-Re SYSTEM AT 1500°C. \*

\*See footnote, Page 15

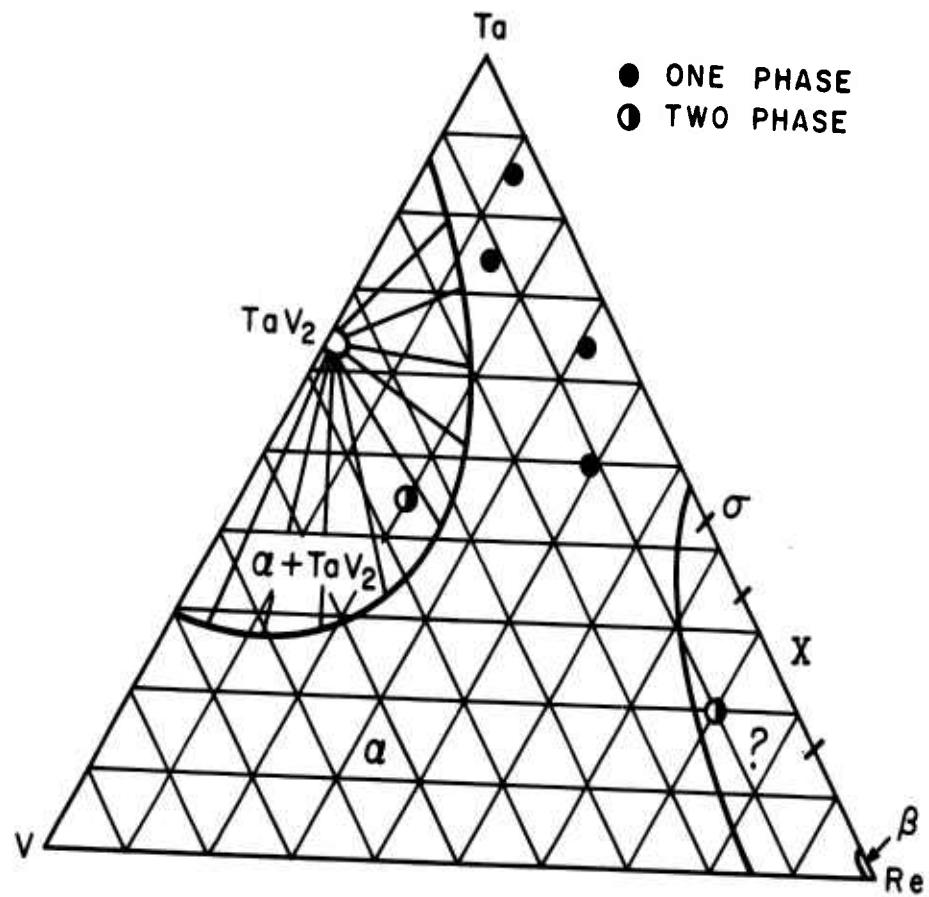


FIG. 56 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-V-Re SYSTEM AT 1000 °C.

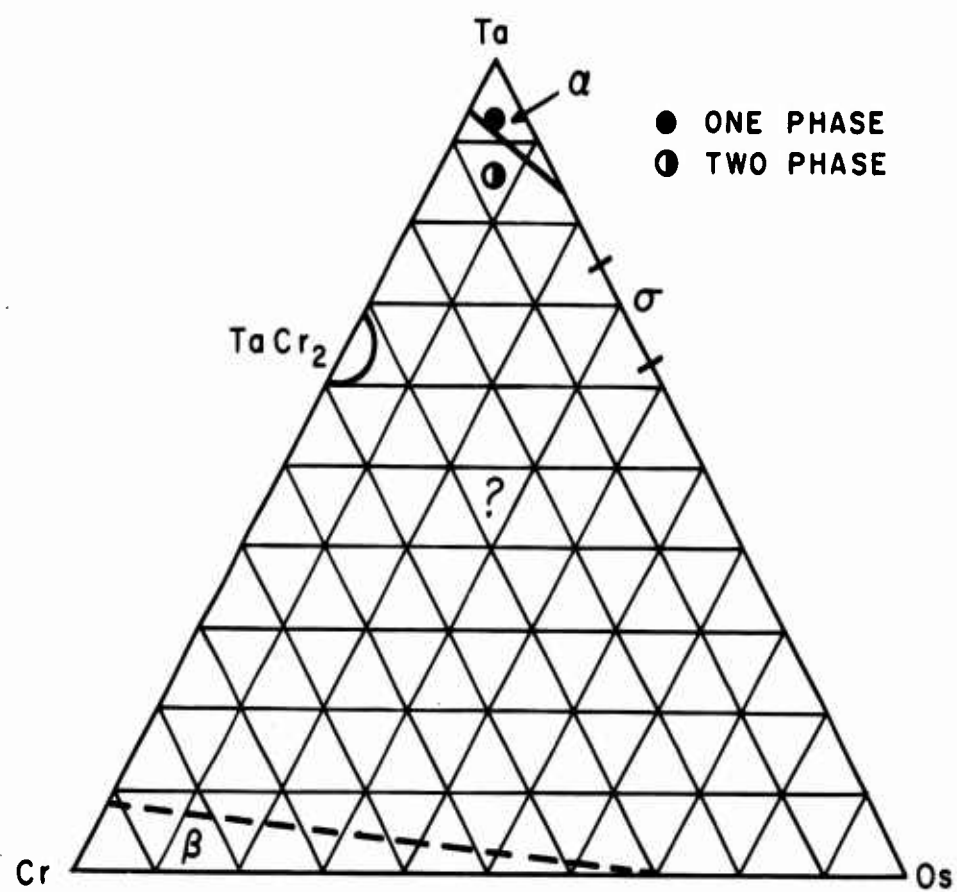


FIG. 57 - TENTATIVE PHASE RELATIONSHIPS  
OF THE Ta-Cr-Os SYSTEM AT 1500° and 1000 °C.

Both alloys were two-phase at the temperatures of interest, and the best approximation of the mutual tantalum-rich solid solubility of these elements is shown in Figure 58.

### 36. Ta-Os-Re System

Four alloys were arc melted:

|        |   |        |   |        |
|--------|---|--------|---|--------|
| 90% Ta | - | 5% Os  | - | 5% Re  |
| 79% Ta | - | 10% Os | - | 11% Re |
| 77% Ta | - | 11% Os | - | 12% Re |
| 64% Ta | - | 5% Os  | - | 31% Re |

These define the limits of the terminal solid solution as shown in Figure 59.

All ternary diagrams were studied to determine if age-hardenability could be postulated for any of the systems. The criterion for hypothesizing, aging was whether or not solubility decreases with decreasing temperature. There is no way of knowing beforehand if the aging propensity of any group of alloys will be significant enough to be commercially attractive. As a general rule, however, the larger the change in solubility the better the chances for significant age hardenability.

In Table III all systems are reviewed and the likelihood of aging is noted. Where complete solid solubility exists (as in W-Ta-Mo) the remarks column indicates "no aging possible". Where solubility changes as a function of temperature are small, the description "aging unlikely" has been used. In all cases, however, experimental work would be required to defend the prediction of age hardenability.

## V. SUMMARY

A cursory survey of thirty-six ternary phase diagrams based on tungsten and/or tantalum together with molybdenum, niobium, osmium, rhenium, vanadium, or chromium was conducted. Prior to studying the solid solubilities in the three-component systems, twenty-eight pertinent binary diagrams had to be established. A thorough review of the literature provided most of these basic data. Where information was not adequate, experimental work with binary alloys was conducted.

All alloys were prepared by conventional arc-melting procedures. Serious difficulties were encountered only with alloys containing chromium. Much volatilization of this metal occurred, and gross changes in composition ensued. Through the judicious use of master alloys and by carefully applying weight loss corrections (after melting), compositions of all alloys including those rich in chromium could be calculated to within





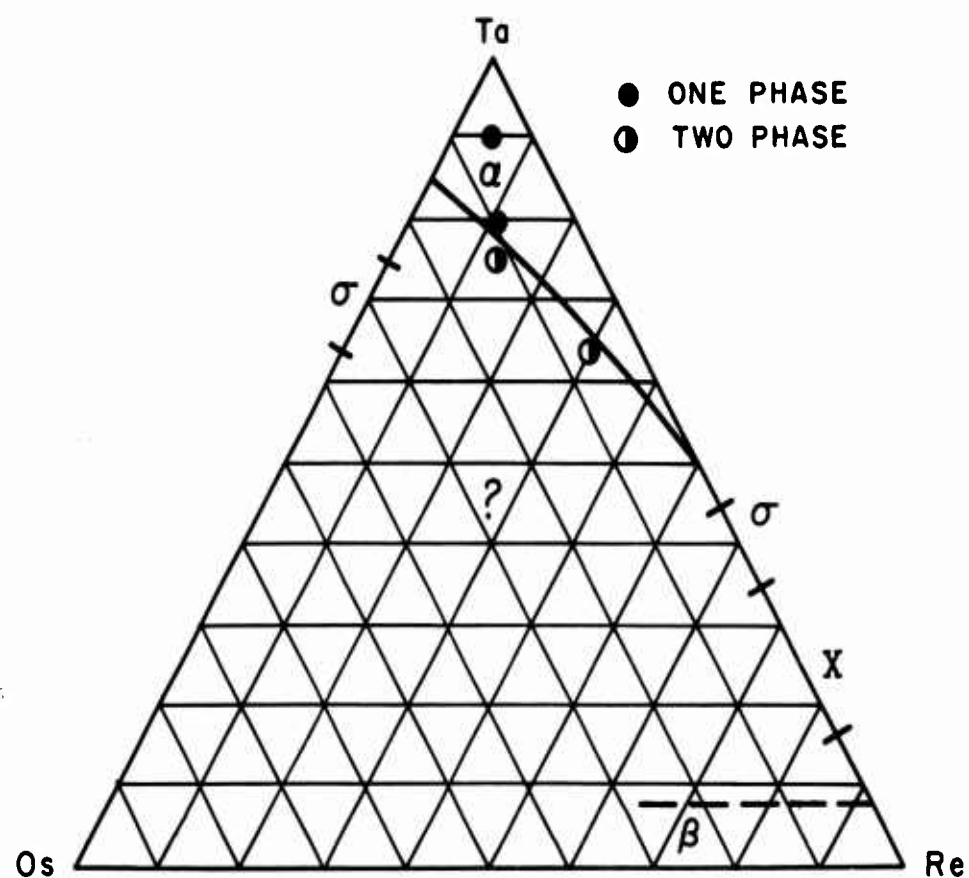


FIG. 59 - PHASE RELATIONSHIPS OF THE  
Ta-Os-Re SYSTEM AT 1500° and 1000°C. \*

\*See footnote, Page 15

TABLE III  
SUMMARY OF POTENTIAL AGE-HARDENABLE  
TERNARY SYSTEMS

| System       | Remarks  |
|--------------|--|
| 1. W-Ta-Mo   | No aging possible  |
| 2. W-Ta-Nb   | No aging possible  |
| 3. W-Ta-V    | Alloys in vicinity of TaV <sub>2</sub> may be age-hardenable     |
| 4. W-Ta-Cr   | Aging possible   |
| 5. W-Ta-Os   | Aging unlikely   |
| 6. W-Ta-Re   | Aging unlikely   |
| 7. W-Mo-Nb   | No aging possible  |
| 8. W-Mo-V    | No aging possible  |
| 9. W-Mo-Cr   | Aging possible   |
| 10. W-Mo-Os  | Aging unlikely   |
| 11. W-Mo-Re  | Aging possible   |
| 12. W-Nb-V   | No aging possible  |
| 13. W-Nb-Cr  | Aging possible   |
| 14. W-Nb-Os  | Aging unlikely   |
| 15. W-Nb-Re  | Aging unlikely   |
| 16. W-V-Cr   | Aging possible   |
| 17. W-V-Os   | Aging unlikely   |
| 18. W-V-Re   | Aging unlikely   |
| 19. W-Cr-Os  | Aging possible   |
| 20. W-Cr-Re  | Aging possible   |
| 21. W-Os-Re  | Aging unlikely   |
| 22. Ta-Mo-Nb | No aging possible  |
| 23. Ta-Mo-V  | Alloys in the vicinity of TaV <sub>2</sub> may be age-hardenable |
| 24. Ta-Mo-Cr | Aging unlikely   |
| 25. Ta-Mo-Os | Aging unlikely   |
| 26. Ta-Mo-Re | Aging unlikely   |
| 27. Ta-Nb-V  | Alloys in the vicinity of TaV <sub>2</sub> may be age-hardenable |

TABLE III (Continued)

| <u>System</u> | <u>Remarks</u>  |
|---------------|---|
| 28. Ta-Nb-Cr  | Aging unlikely  |
| 29. Ta-Nb-Os  | Aging unlikely  |
| 30. Ta-Nb-Re  | Aging unlikely  |
| 31. Ta-V-Cr   | Aging possible  |
| 32. Ta-V-Os   | Alloys in the vicinity of<br>TaV <sub>2</sub> may be age-hardenable |
| 33. Ta-V-Re   | Alloys in the vicinity of<br>TaV <sub>2</sub> may be age-hardenable |
| 34. Ta-Cr-Os  | Aging unlikely  |
| 35. Ta-Cr-Re  | Aging unlikely  |
| 36. Ta-Os-Re  | Aging unlikely  |

1 weight per cent of each component. Selected chemical analyses served to verify the use of weight loss corrections.

Features of the binary diagrams worthy of note include conflicts with existing literature on the position of certain boundaries in the Cr-Re and Nb-Cr and Cr-Os systems. In the work with ternary alloys several generalities can be made concerning phase equilibria. For example, no ternary intermediate phases were found. The presence of  $\text{NbCr}_2$  or  $\text{TaCr}_2$  invariably generated a large two-phase ( $\alpha + \text{XCr}_2$ ) field extending well into the diagram. Severe coring of solid solution alloys was frequently observed. Such structures could easily be interpreted as two-phase configurations, unless the alloys were thoroughly annealed to eliminate coring or specimens were repeatedly repolished and etched to prove the absence of two distinct phases.

The intent in pursuing this study was to provide basic data for future alloy development work in refractory metal systems. Within the broad scope of this program it was not possible to establish phase boundaries definitively. Such accurate phase diagrams determinations can be pursued as the need arises.

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